

1985 PERFORMANCE REPORT

WATER QUALITY SECTION



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1985

PERFORMANCE REPORT

WATER QUALITY SECTION

P.J. Campbell (ed.) Water Quality Section Laboratory Services Branch Ministry of the Environment

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ACKNOWLEDGEMENT

This report is dedicated to the technicians of the Water Quality Section who, in the pursuit of quality data for their customers, performed the more than 600,000 analyses summarized in this report. The magnitude of this task is apparent when one realizes that each datum required analysis, graphical representation, evaluation, and transfer of result to a microcomputer.

and

we gratefully acknowledge the contribution of Laurie Cleary and Michael McVicar who created the software package used to process the QC data and prepare the performance reports, and Ester Yip who assisted in organizing and editing the data.

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The Water Quality Section of the Ministry of the Environment, Laboratory Services Branch is responsible for high production analysis of water quality parameters. By using suitably sensitive instrumentation and methodologies in conjunction with carefully planned and monitored quality control and quality assurance programs, the Water Quality Section is able to maintain a high standard of analytical performance. This performance is certified through regular participation in interlaboratory round-robins. While results on these round-robins (i.e. IJC, USGS, LRTAP) are not included in this report, they are available on request (Appendix A). This report does provide an outline of the Quality Control program, and a summary of performance data for 1985.

In 1985 several tests were discontinued simply because they were requested too infrequently to maintain a QA/QC program. Hence, no data are reported for the filtered total phosphorus test, methylene blue active substances (MBAS) test, volatile acids test and the particulate total carbon (PTC) test. Iron and manganese were transferred to the Inorganic Trace Contaminants Section (ITC) in April 1985, and PTC was later revived by ITC in August 1986. The chlorine test was performed by special request because of its environmental significance but insufficient data are available for this report.

Quality Control Program

The major objective of the Quality Control (QC) program is to ensure immediate detection and correction of unacceptable analytical performance. In practice QC activities are divided into continual checks of basic analytical tools such as chemicals, water purity, containers and daily checks of instrumentation, calibration, recovery.

The quality control program for chemicals involves the purchase of high purity materials, and regular analysis of these chemicals for contamination. An understanding of their shelf lives and health effects is a vital part of this program. Distilled and distilled, deionized water sources are monitored daily for conductivity and dissolved organic carbon. Lines carrying these water supplies are inspected regularly and replaced when necessary. Stability studies for all solutions, whether reagents or standards, are conducted regularly, and the data are utilized to specify shelf-life in method descriptions.

Sample containers, filters, glassware and any other equipment used in the collection and analysis of samples are checked for leaching, adsorption and contamination. The laboratory publication "A Guide to the Collection and Submission of Samples for Laboratory Analysis" (1985) contains recommendations for sample containers, preservatives and sampling techniques.

The first of the daily checks is generally an instrumentation check. Each instrument must be in good working order before an analytical run can commence. A careful record of all maintenance is kept for each workstation.

While a quality control program must encompass all the facets listed above, the greatest effort is probably expended on calibrating the system (procedural step) and checking the calibration (QC). Calibration is achieved using standards covering the analytical range, and is performed before the analytical run commences. Since high degrees of both precision and accuracy are required to detect and minimize any between-run changes, standards are analyzed with as little handling or

preparation as possible. The following steps are taken when setting up most of the analytical instruments.

- Distilled, deionized water (or other appropriate blank) is analyzed to adjust the baseline, or zero setting of the instrument, to a suitable region on the chart recorder or other readout.
- 2) If the system is known to be linear and stable, a high standard at 80% of full scale is analyzed at least twice. When the baseline and high standard have been set, a low standard of 10% -20% of full scale is usually analyzed. An incorrect reading for the latter requires an investigation: the problem may be curvature of the calibration, contamination of the blank water or improper preparation of one of the two standards.
- 3) If the system is known to have a non-linear calibration, several standards are required. A daily calibration curve is developed from these data.

N.B.: Throughout the remainder of this report, the upper concentration of the calibrated range will be referred to as full scale.

Once the system has been calibrated, quality control begins. Depending on the analytical procedure, one or more of the following QC checks is conducted: calibration control, blank control, recovery efficiency, control of potential interferences. To obtain duplicate data, the first aliquots of samples are analyzed early in the run. The second aliquots are analyzed later in the same run. In addition, calibration standards (sensitivity checks) are interspersed throughout the run.

Calibration Control

The calibration is confirmed by means of two control standards QCA and QCB and a long term blank (LTBL) which are made up and maintained independently of the calibration standards. The system is not calibrated with these solutions. QCA and QCB are chosen to be about 70% and 20% of full scale respectively. The long term blank is the water plus any reagent chemicals used in the preparation of QCA and QCB. While working calibration standards may be prepared daily or weekly, the control standards are used for long periods of time. Since the calibration solutions are prepared so often, there is a chance that errors in preparation may occur from time to time. By checking the calibration with control standards, errors in the calibration standards can be detected.

When the control standards are analyzed, their sum (A+B) and difference (A-B) are plotted versus time on a control chart and used immediately by the technician to determine whether the calibration process is in control. The control limits against which the daily A+B and A-B values are compared are determined from practical experience. They should not be set much tighter than the system to which they are applied can tolerate nor should they be set much looser than long term data suggests can be routinely maintained. In general, the daily A+B and A-B values are allowed to vary by \pm 2 to 5% of full scale and \pm 1 to 3% of full scale respectively. If either the sum or the difference is out of control, the system is stopped, corrective action is taken and the control standards are re-analyzed.

The actual values of QCA and QCB are examined whenever the sums or differences are out of control, but they are not themselves separately controlled. Over the long run, the standard deviations of QCA and QCB are used to estimate the

between run standard deviation (S). Values for S and SW can be calculated as follows:

$$2(Sw)^2 = (sd_{A-B})^2$$
 $2S^2 = (sd_A)^2 + (sd_B)^2$

where sdA = standard deviation of QCA in all runs

sdB = standard deviation of QCB in all runs

 sd_{A-B} = standard deviation of QCA-QCB in all runs

N.B.: If a second range is employed for a test, two more QC control standards are used. These are QCC and QCD and are about 80% and 20% for the lower range full scale. (QCB and QCC may be a common solution used for both ranges). The extra pair of calibration controls allow the effect of calibration control concentration over a long term on S and Sw. In many systems, the inrun standard deviation (Sw) is not particularly concentration dependent, although the between run standard deviation (S) may be.

N.B.: For a detailed description of the A/B control process, the reader is directed to references 1,2 and 3 given in the bibliography.

Recovery Checks

In methods where sample preparation such as digestion or extraction is required, a recovery check suitable to that system is required to estimate the efficiency of the analysis. These solutions are not used to calibrate the instrument, but corrections for the digestion blank and matrix effects are estimated and applied if necessary.

Recovery standards are chosen to test all facets of the analysis. If a digestion step is supposed to liberate a substance or convert one substance into another before analysis can take place, then the recovery standard is chosen to test the efficiency of this step. For example, glycine is used to test the efficiency of the digestion step in Total Kjeldahl Nitrogen analyses, and pyrophosphate is used in the Total Phosphorus test. Without pre-treatment (in this case, digestion) these chemicals give a zero response in their respective tests. Recovery standards are usually prepared at 0%, 20%, and 80% of full scale. These recovery standards are then analyzed daily in the same manner as regular samples. The percent recovery can be calculated by:

The 0% standard is referred to as the digested blank and the 80% and 20% standards are referred to as R_1 and R_2 respectively. For an analytical run to be accepted, R_1 and R_2 should be within \pm 5 and 15% respectively of their expected values and the average digested blank should be within three standard deviations of its historical mean.

N.B.: If a second range is employed for a test, at least one additional recovery standard is used (i.e. R3; control limits of ± 5 and 15% of expected values apply to highest and lowest concentrations respectively -see Recovery Plots, p. 10A).

Sensitivity Checks

A gradual increase in peak height of standards may be due to increasing laboratory temperature while gradual decreases in the peak heights may be due to decreasing temperature or deterioration of the reagents. To monitor this, a high standard is run after every twenty samples and the results are compared to the original calibration standards. These checks may be run more frequently where required.

Several tests performed on automated equipment suffer from baseline shift throughout the run due to reagents coating out on the glassware. Contamination of samples also may occur during a run if, for example, an ammonia cleanser is used in the vicinity of the ammonia test. Blanks are interspersed with in run standards to detect any such baseline shifts. The baseline should not shift more than 5% of full scale over the course of the entire run. For most linear systems sensitivity changes within the 5% limits can be corrected mathematically.

Interference Checks

Interference checks are run on any test where a material often may be present in large enough concentrations to affect the results. The checks are usually near the threshold concentration, beyond which the methodological safeguards to minimize the interferences are no longer effective. These checks indicate that the safeguards are effective up to the specified concentration(s).

N.B.: The Water Quality Section does not perform spike checks on a routine basis although they may be included in the method development.

In-Run Duplicate Data

Natural samples are selected on a regular basis for nonadjacent within-run duplicate analysis. Generally, one sample out of twenty is run in duplicate up to a maximum of three per day. One sample of each pair is analyzed near the beginning of the run after QC and Recovery standards. The other sample of the pair is analyzed later in the regular sample run. By analyzing samples in duplicate, the analyst can estimate his ability to obtain repeatable analytical results within a short period of time. The observed differences in duplicate results, for all the duplicates samples, are accumulated and sorted according to sample concentration span. A standard deviation is then calculated for each of these concentration spans using a computer program. The algorithm differs from the conventional standard deviation (designated by a "1" on data summaries) as follows:

Conventional Std. Dev. (1)

$$S_1 = \sqrt{\frac{\sum (x - x_n)^2}{n - 1}}$$

$$S_2 = \sqrt{\frac{\sum_{(x_1 - x_2)_n^2}}{2n}}$$

Where:

sample standard deviationnumber of resultsmean of results

 x_n results from x_1 to x_n Where:

standard deviation of

duplicate samples number of duplicate

pairs

 $(x_1-x_2)_n =$ difference between duplicate **pairs** from

Widely varying results for duplicate analyses of the same sample may signify a breakdown in the analytical system. Duplicate results should differ less than 5% of full scale.

FORMAT FOR PERFORMANCE REPORT

The types of samples analyzed in the Water Quality Section include ground waters, surface waters, sewage, industrial wastes, leachates, soil extracts, drinking water, drinking water sources, and precipitation. In order to handle these varied sample types, the section is divided into a number of laboratories, each responsible for one or more sample types. The Laboratory Information System (LIS) is a centralized computer system which routes samples to the proper laboratory and to a specific workstation.

There is a performance report for each test in each of the laboratories where the test was performed. Information is provided to assist the reader in identifying the data which is appropriate to the various sample types and classes. The performance reports consist of a general summary sheet for each parameter followed by one or more sheets of data tabulated for 1985 and a full page plot of the calibration charts, recovery checks and duplicate results where applicable. The remainder of this section outlines the type of information which is included in the individual performance reports.

SUMMARY SHEET

Title:

This gives the name of the parameter in this particular performance report.

Identification:

Laboratory

This gives the laboratory where the test is performed on the sample types listed below.

LIS Test Name Code

This is the computer code name for the particular parameter, i.e. the code the sampler would see on the final data reports.

Workstation Code

This is the computer code for the workstation to which the sample has been routed.

Method Code

This is the computer code for the analytical procedure which is used at the above workstation.

Method Introduced

This is the date (given as Day/Month/Year) on which this particular method was implemented in the above laboratory.

Units

These are the units in which the results are reported e.g. mg/L as N.

Unit Code

This is the computer code for the units.

Supervisor

This is the name of the supervisor responsible for the laboratory given above.

Sample Type/Matrix

This section lists the various sample types that are accepted at this workstation.

Sampling:

This gives a brief description of the type of bottle to use, what preservatives to use if any, and also the minimum volume of sample that the laboratory requires to do the test. Any sample preparation which must be performed by field personnel is also listed.

Sample Preparation:

This section lists only manual sample preparation techniques (i.e. filtration or extraction) which must be carried out at the laboratory.

Analytical Procedure:

This section gives a brief description of the analytical method used to test the parameter in question. For detailed method descriptions the reader is referred to reference 4 in the bibliography. The methods described in this section apply to 1985.

Instrumentation:

This section gives a brief listing of the instruments used to perform the test. Detailed instrumentation is given in reference 4. Many of the descriptions given will include reference to automated modular continuous flow systems. Such systems consist of an automatic sampler, peristaltic pump, manifold for reagent addition, detection system and an output system such as a chart recorder or printout. The detection system is at least one of the following: colourimeter, scanning spectrophotometer, ion chromatograph, atomic absorption spectrophotometer, conductivity meter, electronic balance, pH meter, or specific ion electrode plus meter. Use of microcomputers to control operation of analytical equipment and/or data acquisition is identified.

Reporting:

This section gives the maximum number of significant figures used to report the result. If an analytical result is determined to be zero, the Water Quality Section reports the MINIMUM INCREMENT (W) in this place. This is done so that the data user can have some idea of how small an analytical result we could determine. For tests using strip chart recorders, which are read manually, W is typically set at 0.5% of full scale. With the increasing use of digital displays and direct computer input, the readability may far outstrip the reproducibility. Here, the W values is trimmed to be one extra significant digit beyond the statistically-determined T value described next.

The DETECTION CRITERION (T) is a value obtained by statistical manipulation of data obtained from the analysis (within run) of duplicate aliquots of the same sample. This value does not guarantee the validity or accuracy of an analytical

result, but it can aid the data user in testing the validity of statements made about a given analytical result or environmental situation. The analyst wishes to warn the data user that a given result is so low that there is a reasonable chance that the value could be zero. If an analytical result greater than the detection criterion is obtained, the risk of making a Type 1 error, i.e. of reporting something as present when in fact it is absent, is less than 0.5%. The detection criterion is determined by multiplying the standard deviation of duplicates in the lowest concentration span by 3. If insufficient data were available, the T value was calculated using the standard deviation from the next higher concentration span.

Calibration:

This section lists the number of standards used to calibrate the analytical system daily.

Controls:

This section lists the control checks used throughout the analytical process. The CALIBRATION control standards QCA, QCB, QCC and QCD and LTBL are used to monitor the calibration. The RECOVERY controls are used to monitor the efficiency of the overall test. These are the $R_1,\,R_2,\,R_3,\,R_4$ and digested blank standards.

The DRIFT controls are the standards which are used to monitor the instrument stability throughout a run. The number of standards used is given and the frequency of their usage is given whenever the latter differs from "every 20 samples".

The INTERFERENCE controls are those solutions which are analyzed to check that potential interferences are eliminated. Concentrations of the interference controls are designed to exceed concentrations expected in the vast majority of samples

Modifications:

This section lists any modifications to the test since the publication of "Handbook of Analytical Methods for Environmental Samples" (HAMES) (Reference 4).

NOTES:

Any explanatory notes which the analyst feels may aid the data user in interpreting the information provided.

N.B.: If headings have been omitted from a particular summary sheet, the reader should assume that such headings are not applicable to this test.

PERFORMANCE DATA

For each performance report there will be at least one tabulated data page to cover 1985.

Title:

This gives the name of the parameter in this particular performance report.

Quality Control Data From:To:

These dates specify the collection period for the data tabulated on this page. Dates are given as day/month/year. Pages covering portions of the year are provided if any of the control solutions were changed or the method was modified. QC data are not provided for parts of the year when the test was inactive.

Lab:

This lists the laboratory in which this data was collected.

Analytical Range:

The analytical range in concentration units is given. The range normally covers the span from the detection criterion to 100% of full scale. N/A or a blank for the detection criterion indicates that insufficient data were available in the lowest range of duplicates for direct calculation.

Calibration Control:

This section tabulates all the data collected for the calibration control standards (QCA, QCB, QCC and QCD). The table headings give the number of data points collected, the expected concentration for each control standard as well as their sum and difference; the average concentrations measured, the bias (difference obtained by subtracting the expected concentration from the average concentration measured); and the standard deviation calculated for each standard, their sum and their difference.

The within run standard deviation (Sw) and the between run standard deviation (S), the ratio S/Sw and the ranges for acceptance of the day's A+B and A-B values are also shown.

- N.B.: If the system employs two analytical ranges, values for QCC and QCD are also given.
- N.B.: All data are reported in the concentration units shown at the top of the page unless otherwise stated in the Summary Sheet.

Recoveries:

This section tabulates the data collected for the recovery control standards (R_1 , R_2). The number of data points, the expected concentration, the average measured concentration and the calculated standard deviation are listed for each recovery standard. If no recovery checks were performed, this section is omitted.

N.B.: All data are reported in the concentration units shown at the top of the page unless otherwise stated.

Duplicates:

This section tabulates the data collected from the analyses (within run) of duplicate aliquots from the same sample. The number of data pairs used, the sample concentration spans into which the analyses were sorted, the mean standard deviation and the coefficient of variation (relative standard deviation) for each

span are given. The coefficent of variation (%) is obtained by dividing the mean standard deviation for a particular span by the mean concentration of duplicate results in that span and then multiplying by 100.

N.B.: All data are reported in the concentration units shown at the top of the page unless otherwise stated.

Detection Criterion:

The detection criterion, which is based on duplicate data, is calculated by multiplying the mean standard deviation for the lowest concentration span by 3. In some years the amount of such data is insufficient, and thus the calculated detection criterion is suspect.

Other Checks:

This section lists data for checks such as the long term blank, digested blank, standard cal settings on colourimeters, etc. The number of data points, the data mean and the calculated standard deviation are given.

QUALITY CONTROL GRAPHS:

For each data page there is one QC graph for 1985.

Title:

This gives the name of the parameter in the performance report and appropriate units.

Dates:

Dates on plots correspond to identical periods on the performance data pages (day/month/year).

Calibration Control:

A+B, A-B, C+D, and C-D (if used) are plotted on a horizontal scale covering the period of data collection. Points are equally spaced. The vertical scale is centred on the expected value for calibration control. Control limits (+CL) were chosen from previous analytical performance (when available). Frequent excursions outside the limits indicate a system out of control or excessively tight limits. An asterisk marks each point more than 15% outside the limits. Units on the vertical scale are identical to those in the title unless marked otherwise in the Summary Sheet.

Recovery Plots:

Where recovery checks are performed the highest and lowest concentrations are plotted (i.e. if four recovery checks are run, R1 and R4 are plotted). The horizontal scale is identical to the calibration control plots. The vertical scale is centred on the expected value for the recovery checks. Arbitrary control limits have been assigned (but not drawn) as ± 5 and 15% of the expected value for two recovery checks, ± 5 , 10, 15% for three checks, and ± 5 , 8, 12, 15% for four checks

going from highest to lowest concentration. The sliding scale is used since recoveries usually range from 80% to 20% of full scale. Hence, the absolute control limits are about + 4% at the high and + 3% at the low end of full scale.

Duplicate Plots:

All duplicate results for the period are summarized in a three segment plot at the bottom of the page. The three segments are 0-20%, 20-50% and 50-100% of full scale (full scale is defined below on the same page and may be different from the value given in the Performance Report to improve the clarity of presentation). In each concentration category, the absolute differences between duplicate samples are tabulated to determine the frequency with which the duplicate pairs differ by 0-2, 2-4, 4-6, and greater than 6% of full scale. The relative number of occurances in each range are plotted as a percentage of the total in three frequency histograms. As the histogram ranges are calculated with respect to full scale, they cover the same span of absolute differences in concentration for each of the graphs, and the distributions can therefore be compared directly.

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APPENDIX - A

Responsibility for QC/QA Tasks: 1985

The supervisors for the Water Quality Chemistry laboratories (416 248 3512) and their unit leaders or senior technicians in 1985 were:

Manager - Larry Vlassoff Domestic Water Laboratory - Michael Rawlings - Stuart Barnes Precipitation Laboratory - Michael Rawlings - Jenifer McBride Rivers and Lakes Laboratory - Joan Crowther - Walter Wright - Stella Tracy - Abraham "Pete" Millar Sewage and Industrial Waste - Peter Campbell Laboratory - Ben Cheung - Vera Turner Dorset and Mobile Laboratory - Frank Tomassini - Charlie Chun (Dorset) (705 766 2412) - Peter Wilson (Mobile)

Responsibility for QC/QA Tasks: 1986

Reorganization of staff in April 1986 led to the following changes in responsibility:

Class	Contact & Assistance			
С	Chlorophyll	P. Millar/B. Bobor		
G	Great Lakes	J. Crowther/H. Broomer		
Н	Public Health Insp.	J. Crowther/V. Ferraro		
R	The state of the s			
S P	Precipitation	J. McBride/		
W	Water (Grand/Domestic)	J. McBride/		
AS	APIOS Air Filters	J. McBride/		
I & LW	Industrial Wastes	S. Tracy/V. Turner		
SX	Soil Leach (APIOS)	J. Crowther/J.McBride		
CC & ERTF	High Priority	V. Turner/B. Cheung		
DP, DR, DX, DS	Dorset Precipitation, Rivers & Lakes,	C. Chun/S. Barnes		
,	Soil Leach, Stemflow	P. Grauds D. Evans		

Supervisors (Contact for General Discussion, Planning, Problem Samples, etc.)

Mike F	Pawlings	Colo	urimetri	Tests				
Frank	Tomassini	Ion (Chromato	graphic	and	Atamic	Absorption	Tests
Peter	Campbell	BOD,	Solids,	Matrix	Asse	ssment,	Titration	Tests

Joan Crowther All tests

APPENDIX - B

Glossary

AAS - Atomic Absorption Specrophotometer

Abs - Absorbance Av - Average BL - Blank

C - Degrees Centrigrade

cm - Centimeter

Concn - Concentration

Date - Day/month/year

DDW - Distilled, deionized water

DW - Distilled water

FTU - Formazin Turbidity Units

g - Gram

HAMES - Handbook of Analytical Methods for Environmental Samples, M.O.E.

HOAC - Acetic Acid
HZU - Hazen Units

L - Liter

LAB - Laboratory

LIS - Laboratory Information System

LTBL - Long Term Blank

M - Molar mg - Milligram

mil - One-thousands of an inch

min - Minute
mL - Milliliter
mm - Millimeter
N - Normal

N/A - Not Available nm - Nanometer

oz - Ounce

QC - Quality Control

rpm - Revolutions per minute

Between run standard deviation for QC

APPENDIX - B (Continued)

Glossary

Sw - Within run standard deviation for QC

sd - Standard deviation

S. class - Weights that have not been officially calibrated within past six months

Standard Cal - Colourimeter setting controlling electronic expansion

STD - Standard

TCU - True Colour Units

u - Micron or Micrometer

us - Microgram uS - Micro-Siemen

V/V - Concentration based on volume measurements

APPENDIX - C

W & T:

The latest printout (valid at date of publication) of W & T values for all known active workstations in the Water Quality Section follows. Since the values changed after the last QA/QC Performance Report (1982-84) and are in the process of changing again, clarification is needed.

Workstation, test codes, and full scale have been defined earlier in the introduction. Std. dil. refers to the standard dilution employed in sample preparation. A standard dilution of "I" indicates that the sample was analyzed without dilution with respect to the standard used for calibration. The 1985 W and T values and the standard deviation of duplicates (S Dupl) were obtained from the 1985 performance data in this report, when available. Some of the values are based on 1986 data. Parameters/workstations/test codes reflect the reorganization of the Water Quality Section in early 1986.

Prior to 1985, W was the minimum determinable amount, and T was 1.645 times the standard deviation of duplicates in a concentration range of about 0-20% of full scale. The W value was supplied to the client to indicate the smallest amount that we could determine when the actual response (=result) was zero. In 1985, T was made three times the standard deviation of duplicates (based on the 1982-84 Performance Report -see reference 3) and W was changed only where the minimum amount had changed. The increase in T was made to be consistent with recommendations by the American Chemical Society (see reference 8) and to provide a level above which data users could have more than 99% confidence that a result obtained was above zero.

Through internal discussions and attempts to provide a consistent approach to data reporting, the Water Quality Section has been asked to calculate W from the standard deviation of duplicates by rounding down the nearest 1,2 or 5 digit. T is to be five times W. The new list has been calculated this way where possible, using the S Dupl values.

Although values below W will still be treated as zero, we will retain an extra digit in all results greater than or equal to W so that the standard deviation of duplicates can be calculated, and we can detect small improvements or deterioration in the performance of tests. The introduction date for the new W and T values has been set for November 1, 1986.

PARAMETER	UNITS	WORKSTATIONS	TEST CODES	FULL SCALE	STD	1985 W	1985 T	S DUPL	NEW W	NEW T
Acidity-Gran	ueq/L as H	PHACD	ACDG	1000	1	0.1	4.7	1.56	1	5
Acidity-TFE	mg/L as CaCO3	PHACD	ACDT	100	1	0.01	0.24	0.081	0.05	0.25
Alkalinity-Gran		DOT	ALKTI	25	1	0.01	0.24	0.078	0.05	0.25
Alkalinity-Gran	mg/L as CaCO3	RATS	ALKTI	25	1	0.01	0.45	0.15	0.1	0.5
Alkalinity-TFE	mg/L as CaCO3	DOT	ALKT, ALKT3	100	1	0.01	0.254	0.081	0.05	0.25
Alkalinity-TFE	mg/L as CaCO3	WATS	ALKT	500	1	0.2	1.6	0.55	0.2	1.0
Alkalinity-TFE	mg/L as CaCO3	RATS	ALKT	250	1	0.01	0.78	0.261	0.2	1.0
Aluminum	ug/g as Al	DOCATION	ALESC	200	1	0.1	3.9	1.3	1	5
Aluminum	% as Al	DOMETDI	ALED1	0.4		0.001	0.006	0.002	0.002	0.10
Aluminum	% as Al	DOMETALX	ALEPY	0.2		0.001	0.009*		0.002	0.010
Aluminum	ug/g as Al	DOSOLAL	ALECA	40	1	0.01	0.72	0.24	0.2	1.0
Aluminum-CVreac.	ug/L as Al	DOMISC	ALNDCV, ALEXCV	1000	1	1	12#	4.0	2	10
Aluminum	ug/L as Al	DOAAS	ALUT	200	1	1	3#	1	1	5
Cadmium	ug/L as Cd	DOAAS	CDUT	2	1	0.001	0.057#		0.01	0.05
Calcium	ug/g as Ca	DOCATION	CAEXC	800	1	1	12	4	2	10
Calcium	mg/L as Ca	WAAS	CAUR	175	1	0.1	0.9	0.28	0.2	1.0
Calcium	mg/L as Ca	PRAA	CAUR	2	1	0.01	0.08	0.025	0.02	0.1
Calcium	mg/L as Ca	RMAAS	CAUR	35	1	0.01	0.33	0.11	0.1	0.5
Carbon-Carbonate	% as C	DOTIC	TIC	100	1	1	22	7.4	5	25
Carbon-Dis.Inor.	mg/L as C	DOCOP	DIC	10	1	0.01	0.05	0.017	0.01	0.05
Carbon-Dis.Inor.	mg/L as C	ROM	DIC	40	1	0.2	0.46	0.154	0.2**	1.0
Carbon-Dis.Org.	mg/L as C	ROM	DOC	20	1	0.1	0.28	0.095	0.1**	0.5
Carbon-Soil Org.	% as C	DOOXMAT	ORGC	100	1	0.01	0.25	0.084	0.05	0.25
Chloride	mg/L as Cl	PRICL	CLIDUR	2	1	0.01	0.05	0.018	0.01	0.05
Chloride	ug/filter as Cl	PRLOV	CLIDUR	100	1	0.5	0.8	0.28	0.5**	2.5
Chloride	mg/L as Cl	ROM	CLIDUR	50	1	0.05	0.24	0.081	0.05	0.25
Chlorophyll-a	ug/L	RCHLO	CHLRAT	10	1	0.01	0.51	0.171	0.1	0.5
Chlorophyll-b	ug/L	RCHLO	CHLRBT	10	1	0.01	0.31	0.102	0.1	0.5
Chlorophyll-acid		RCHLO	CHLRAC	10	1	0.01	0.43	0.144	0.1	0.5
Colour-true	TrueColourUnits	DOCC	COLITR	100	1	1	4	1.2	1	5
Colour-true	TrueColourUnits	WCOL	COLITR	100	1	0.5	1.1	0.37	0.5**	2.5
Colour-true	TrueColourUnits	ROCOL	COLITR	100	1	0.1	1.6	0.53	0.5	2.5
Conductivity	us/cm	WATS	COND25	5000	1	1	3	1.0	1	5
Conductivity	us/cm	DOCC	COND25	300	1	0.1	1.3*	0.43	0.2	1
Conductivity	us/cm	PRIC1	COND25	100	1	0.1	0.9	0.31	0.2	1
Conductivity	us/cm	RATS	COND25	2000	1	1	2.1	0.7	1**	5
Conductivity	us/cm	COND-SEW	COND25	3000	1	1	4	1.2	1	5

PARAMETER	UNITS	WORKSTATIONS	TEST CODES	FULL SCALE	STD	1985 W	1985 T	S DUPL	NEW W	NEW T
Copper	ug/g as Cu	DOHMTE	CUUT	250	1	0.1	1.6	0.55	0.5	2.5
Fluoride	ug/L as F	DOSPF	FFIDUR	70	1	0.1	1.4	0.48	0.2	1.0
Fluoride	mg/L as F	WFNO3	FFIDUR	2	1	0.01	0.03	0.009	0.01**	0.05
Iron	% as Fe	DOMETDI	FEED1	0.4		0.001	0.009	0.003	0.002	0.010
Iron	%as Fe	DOMETALX	FEEPY	0.2	1	0.001	0.006*		0.002	0.010
Lead	ug/g as Pb	DOHMTE	NIUT	250	1	0.1	2.0	0.7	0.5	2.5
Lead	ug/L as Pb	DOASV	PBUT	2	1	0.01	0.6*	0.2#	0.1	0.5
Magnesium	ug/g as Mg	DOCATION	MGESC	300	1	0.1	2.8	0.92	0.5	2.5
Magnesium	mg/L as Mg	WAAS	MGUR	35	1	0.05	0.3	0.11	0.1	0.5
Magnesium	mg/L as Mg	PRAA	MGUR	0.5	1	0.005	0.012	0.0041	0.005**	0.025
Magnesium	mg/L as Mg	RMAAS	MGUR	7	1	0.01	0.06	0.021	0.02	0.1
Nickel	ug/g as Ni	DOHMTE	NIUT	250	1	0.1	2.0	0.7	0.5	2.5
Nitrogen A+A	ug/L as N	DONUT	NNHTFR	1000	1	1	3.5	1.17	1	5
Nitrogen A+A	mg/L as N	PRNUT	NNHTUR	5	1	0.005	0.014	0.0039	0.005**	0.025
Nitrogen A+A	ug/filter as N	PRSEQ	NNHTFR	125	1	0.125			0.1	0.5
Nitrogen A+A	mg/L as N	RNDNP	NNHITR	2	1	0.002	0.01	0.0037	0.002	0.010
Nitrogen A+A	mg/L as N	SDNP	NNHTFR	50	1	0.05	0.15	0.052	0.05	0.25
Nitrogen-NO3	mg/L as N	PRICL	NNO3UR	2	1	0.01	0.05	0.018	0.01	0.05
Nitrogen-NO3	ug/filter as N	PRSEQ	NNO3FR, NNRICF	50	1	0.25	0.5	0.18	0.2	1
Nitrogen-NO3	ug/filter as N	PRLOV	NNO3UR	100	1	0.5	1.2	0.41	0.5**	2.5
Nitrogen-NO3+NO2		WFNO3	NNOTUR	20	1	0.1	0.1	0.03	0.1**	0.5
Nitrogen-NO3+NO2		DONUT	NNOTER	500	1	2	9	3.0	.2	10
Nitrogen-NO3+NO2		RNDNP	NNOTFR	5	1	0.005	0.06	0.02	0.02	0.10
Nitrogen-NO3+NO2		SDNP	NNOTFR	50	1	0.05	0.24	0.08	0.05	0.25
Nitrogen-NO2	mg/L as N	RNDNP	NNO2FR	0.25	1	0.0005	0.003	0.001	0.001	0.005
Nitrogen-NO2	mg/L as N	SDNP	NNO2FR	2	1	0.005	0.009	0.0031	0.005**	0.025
Nitrogen-T.Kjel.		RINP	NNTKUR	2	1	0.01	0.06	0.019	0.02	0.10
Nitrogen-T.Kjel.		STKNP	NNTKUR	25	2	0.05	0.19	0.063	0.05	0.25+
Oxygen-Biochem.	mg/L as O	SBBOD, SBBOD5	BOD5, BOD5C, ETC	400	1	0.01	1.0	0.33	0.2	1.0
Oxygen-Chem. Dem.		RCOD	COD, CODF	100	1	1	2	0.5	1**	5
Oxygen-Chem. Dem.		SBCOD	COD, CODF	500	1	2	13	4.4	2	10
Oxygen -diss.	mg/L as O	DOCOP	DO		1					
Particle size	% sand	DOPARTZ	SAND	100	1	0.1	2.5	0.84	0.5	2.5
Particle size	% silt	DOPARTZ	SILT	100	1	0.1	3.4	1.2	1	5
Particle size	%clay	DOPARTZ	CLAY	100	1	0.1	4.0	1.3	1	5
TOTAL DIE	00201									

PARAMETER	UNITS	WORKSTATIONS	TEST CODES	FULL SCALE	STD	1985 W	1985 T	S DUPL	NEW W	NEW T
pH p	OH units	DOCOP	PH	-	1	0.01	N/A			
	oH units	DOT	PH	_	1	0.01	N/A			
pH p	oH units	DOSOILPH	PHEW, PHECA	-	1	0.01	N/A			
	oH units	WATS	PH	-	1	0.01	N/A			
pH p	oH units	PHACD	PH	-	1	0.01	N/A			
pH r	oH units	RATS	PH	-	1	0.01	N/A			
	oH units	SBPH	PH	-	1	0.01	N/A			3.0
Phenolics-Reac. u	ug/L as Phenol	ROPHEN	PHNOL	50	1	0.2	1.0	0.35	0.2	1.0
Phosphorus-Ortho m	mg/L as P	RNDNP	PPO4FR	0.125		0.0005		0.0009	0.0005	0.0025
Phosphorus-Ortho m	mg/L as P	SDINP	PPO4FR	10	1	0.01	0.05	0.017	0.01	0.05
Phosphorus-Total u	ug/L as P	DOP	PPUT1,_2,_3	200	1	0.1	0.8	0.28	0.2	1.0
Phosphorus-Total m	mg/L as P	RINP	PPUT	0.2	1	0.001	0.007	0.0023	0.002	0.010
Phosphorus-Total m	mg/L as P	STKNP	PPUT	5	2	0.01	0.03	0.0086	0.01	0.05+
Potassium u	ug/g as K	DOCATION	KKESC	300	1	0.1	6.6	2.2	2	10
Potassium n	mg/L as K	WAAS	KKUR	50	1	0.05	0.15	0.051	0.05	0.25
	mg/L as K	PRAA	KKUR	1	1	0.005	0.025	0.0082	0.005	0.025
Potassium u	ug/Filter as K	PRLOV	KKUR	50	1	0.25			0.2	1
Potassium n	mg/L as K	RMAAS	KKUR	10	1	0.01	0.05	0.017	0.01	0.05
Silicon n	mg/L as Si	ROM	SIUR	10	1	0.02	0.04	0.012	0.02**	0.10
Sodium n	mg/L as Na	WAAS	NAUR	100	1	0.1	0.7	0.23	0.2	1.0
Sodium r	mg/L as Na	PRAA	NAUR	1	1	0.005	0.016	0.0053	0.005	0.025
Sodium ı	ug/Filter as Na	PRSEQ	NAUR	50	1	0.25	2.40	0.80	0.2	1
Sodium r	mg/L as Na	RMAAS	NAUR	20	1	0.01	0.08	0.026	0.02	0.10
Solids-Diss. r	mg/L	SOLIDS	RSF	3000	1	0.5	9.2	3.06	2	10
Solids-Diss.Ign. r	mg/L	SOLIDS	RSFA	3000	1	1	6.0	1.99	2	10
Solids-Partic. n	mg/L	SOLIDS	RSP	3000	1	0.1	2.5	0.83	0.5	2.5
	mg/L	SOLIDS	RSPA	3000	1	0.1	N/A	0.18	0.5	2.5
Solids-Total	mg/L	SOLIDS	RST	6E04	1	0.5	13	4.4	2	10
Solids-Tot.Ign. r	mg/L	SOLIDS	RSTA	3E04	1	0.5	52	17.2	10	50
	ug/g as SO4	DOANIONX	SSO4EW	100	1	0.1	2.6	0.9	0.5	2.5
	mg/L as SO4	PRIC1	SSO4UR	10	1	0.05	0.09	0.032	0.05**	0.25
Sulphate 1	ug/filter as SO4	PRSEQ	SSO4FR, SSO4NF	250	1	1.25	3	1.1	1	5
	ug/filter as SO4	PRLOV	SSO4UR	500	1	2.5	9	3.1	2	10
	mg/L as SO4	RMDSO4	SSO4UR	200	1	0.05	0.76	0.253	0.2	1.0
Sulphur Dioxide 1	ug/filter as SO2	PRSEQ	SSO2FR	350	1	1.65	3.9	1.28	1	5

PARAMETER	UNITS	WORKSTATIONS	TEST CODES	FULL SCALE	SID	1985 W	1985 T	S DUPL	NEW W	NEW T
Turbidity Turbidity Zinc Zinc	FIU FIU ug/L as Zn ug/g as Zn	WIURB RMIURB DOASV DOHMIE	TURB TURB ZNUT ZNUT	200 50 15 250	1 1 1	0.01 0.01 0.01 0.1	0.09 0.21 0.71# 4.0	0.031 0.069 0.24 1.3	0.02 0.05 0.2	0.10 0.25 1.0 5

^{*} Standard deviation of duplicates (S DUPL) not taken from lowest range (insufficient data).

** New "W" was rounded up to be above smallest determinable value (1985 W).

^{# 1986} data used.

⁺ Minimum dilution factor is 2. W & T given above are values for undiluted samples (factor 1).

*** ACIDITY - GRAN ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/08/82

LIS Test Name Code: ACDG Units : ueq/L as H

Work Station Code : PHACD Unit Code : 063801
Method Code : 0018T5 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

Sample aliquots(10.0 mL) are titrated with 0.01N sodium hydroxide to a pH >8.3. The titrant is standardized against 0.0005N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis.

N.B. pH and total fixed endpoint acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.1

Detection Criterion (T): 4.7

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL (expected result is 16.6 ueq/L as H) plus two standards, eg, QCA

MODIFICATIONS:

01/08/82- QC program was expanded to include Gran acidity for which the reporting units are ug/L as H.

01/05/83- System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

01/06/84- Normality of KHP used to standardize the base was reduced from 0.005N to 0.0005N

ACIDITY - GRAN QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 4.7 to 1000 ueq/L as H

			COLPOSI .	
CAL	IBKHI	IUN	CONTROL:	

Deviation	
6.5	
3.5	
9.2	
5.0	
	3.5 9.2

s.d.(AB): Sw(within run): 3.5 S(between runs): 5.2 S/Sw: 1.48

On any given day the calibration is accepted if the values obtained lie within the ranges:

655 to 745 for A+B 270 to 330 for A-B

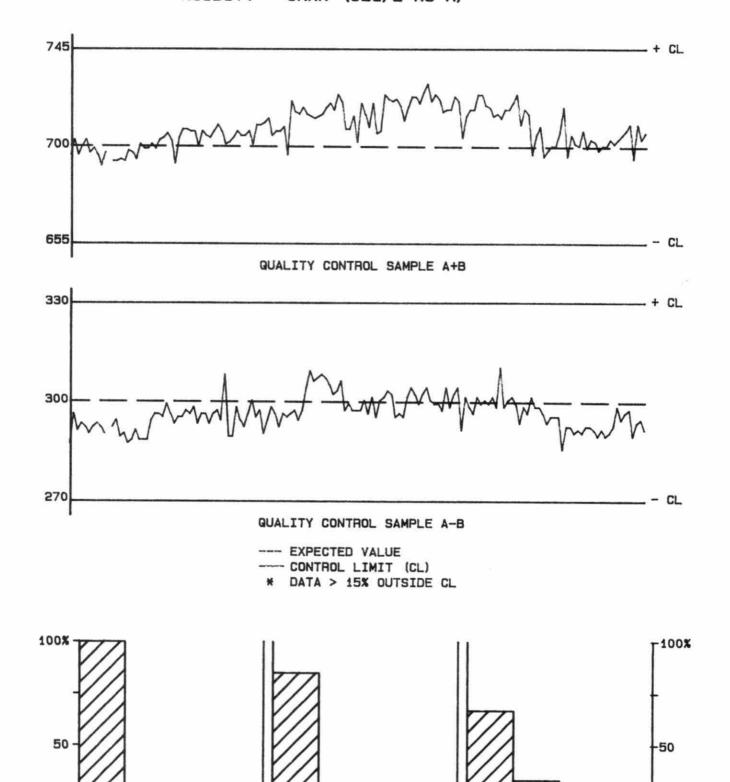
DUPLICATES:	Number of	S	a.mp	le	Mean(2)	Coefficient
	Data Pairs	Cond	n	Span	s.d.	of var.(%)
	112	0.0	-	40.0	1.56	6.1
	169	40	-	100	2.2	3.2
	77	100	-	250	3.2	2.4
	14	250	-	500	16.9	5.2
	3	500	-	1000	9.3	1.5
	375	0	ver	all	4.0	N/A

DETECTION CRITERION: 4.7

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	148	16.03	2.677

QUALITY CONTROL GRAPHS ACIDITY - GRAN (UEQ/L AS H)

FROM: 02/01/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UEQ/L AS H

20-50% F.S.

4-6%

2-4%

50-100% F.S.

4-6%

2-4%

0-2%

2-4% 4-6%

0-20% F.S.

> 6%

0-2%

*** ACIDITY - TOTAL FIXED ENDPOINT (TFE) ***

IDENTIFICATION:

Laboratory : Dorset

Method Introduced: 16/08/82

Supervisor

: F. Tomassini

Units : mg/L as CaCO3

Sample Type/Matrix: Streams, Precipitation, Groundwaters

SAMPLING:

Quantity Required: 25 mL

Container : Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.01 N sodium hydroxide to a pH endpoint of 8.3. The titrant is standardized against 0.005N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

N.B. pH and Gran acidity are determined simultaneously.

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.52

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA
Drift : 2 standard buffers -4 times daily.

ACIDITY - TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 03/01/85 TO 18/12/85

Lab: Dorset

Analytical Range: 0.52 to 50.0 mg/L as CaCO3

CAL	TRRA'	TION	CON	TROL
UnL	TOKU	LIUIA	CUIT	I KUL .

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	96	25.00	25.10	0.10	0.275
b		95	10.20	10.10	-0.10	0.229
a+b	:	95	35.20	35.20	0.00	0.361
a-b	1	95	14.80	14.99	0.19	0.356

s.d.(AB): Sw(within run): 0.252 S(between runs): 0.253 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

32.95 to 37.45 for A+B 13.30 to 16.30 for A-B

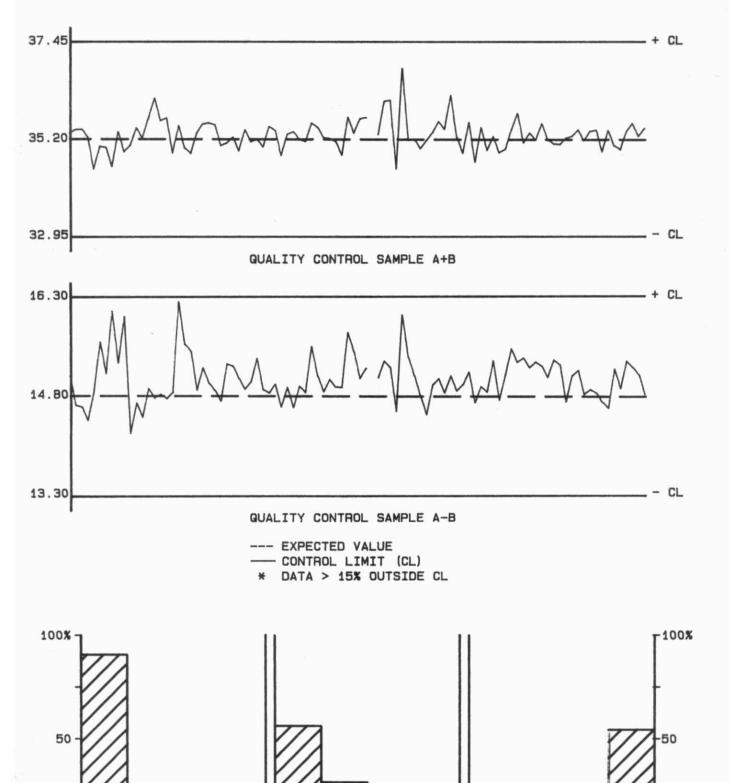
DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	19	0.00 - 2.00	0.174	12.1
	98	2.00 - 5.00	0.266	7.2
	67	5.00 - 10.00	0.816	11.7
	55	10.0 - 25.0	1.07	6.9
	10	25.0 - 50.0	2.19	6.4
	249	Overall	0.81	N/A

DETECTION CRITERION: 0.52

OTHER CHECKS:	Num	ber Data	Standard(1)
	of D	ata Mean	Deviation
Long Term Blank	: 85	0.14	0.201

QUALITY CONTROL GRAPHS
ACIDITY - TOTAL FIXED ENDPOINT (MG/L AS CACO3)

FROM: 03/01/85 TO: 18/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS CACO3

20-50% F.S.

4-6%

4-6%

50-100% F.S.

2-4%

2-4%

0-20% F.S.

*** ACIDITY - TOTAL FIXED ENDPOINT (TFE) ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/05/79

LIS Test Name Code: ACDT Units : mg/L as CaCO3

Work Station Code : PHACD Unit Code : 064915

Method Code : 001BT2 Supervisor : M. Rawlings Sample Type/Matrix : Precipitation, Throughfall, Stemflow, Domestic Waters,

Rivers, Lakes, (by special request: Industrial Waste, Sewage)

SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated in an automated system with 0.01N sodium hydroxide to a pH endpoint of 8.3. The titrant is standardized by titrating 0.0005N potassium hydrogen phthalate to the pH endpoint of 8.3. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. N.B. pH and Gran acidity are determined simultaneously

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.24

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL plus two standards, eg, QCA

MODIFICATIONS:

01/04/82- Sample volume was decreased from 100.0 to 10.0 mL.

01/05/83- System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

01/06/84- Normality of KHP used to standardize base was reduced from 0.005N to 0.0005N.

NOTES:

Due to the instability of the QC standards at these concentration levels, calibration control limits are based on measured averages rather than theoretical concentrations.

ACIDITY - TOTAL FIXED ENDPOINT (TFE) QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 0.24 to 100.0 mg/L as CaCO3

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	148	24.9	25.1	0.2	0.32
ь	2	148	10.7	10.3	-0.4	0.16
a+b		148	35.6	35.4	-0.2	0.44
a-b	:	148	14.2	14.8	0.6	0.26

s.d.(AB): Sw(within run): 0.18 S(between runs):

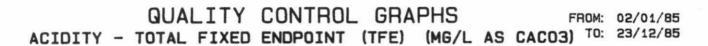
0.25 S/Sw: 1.38

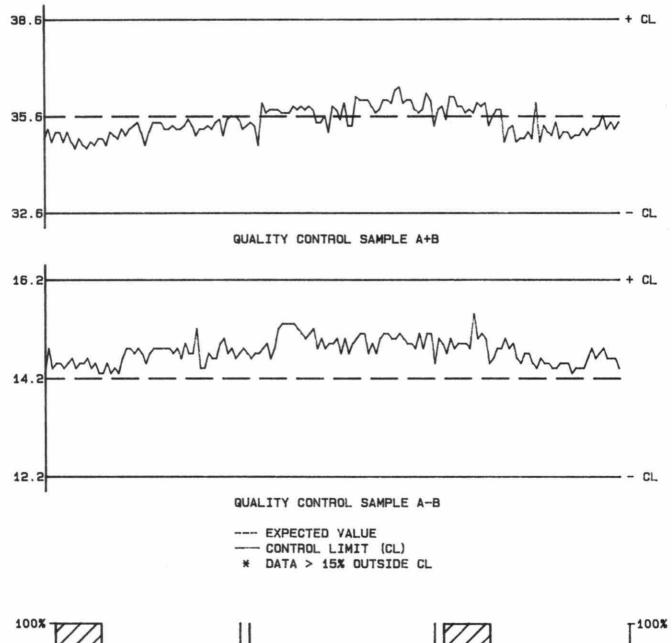
On any given day the calibration is accepted if the values obtained lie within the ranges:

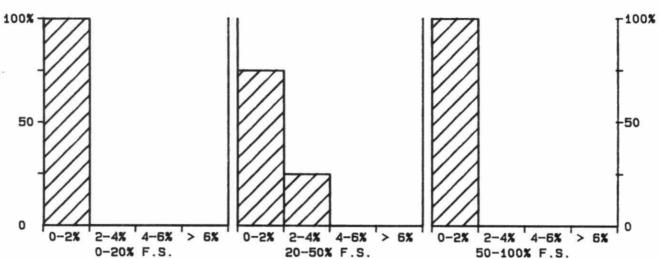
> 32.6 to 38.6 for A+B 12.2 to 16.2 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
			5.0.	01 Var.(7.)
	108	0.00 - 2.00	0.081	6.0
	168	2.00 - 5.00	0.099	2.9
	76	5.00 - 10.00	0.124	1.9
	20	10.0 - 25.0	0.75	5.1
	4	25.0 - 100.0	0.38	0.9
	376	Overall	0.20	N/A

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 148	0.84	0.134







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS CACO3

*** ALKALINITY - GRAN ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced: 28/07/78

Supervisor

: F. Tomassini

Units : mg/L as CaCO3

Sample Type/Matrix: Streams, Lakes, Precipitation, Groundwaters

SAMPLING:

Quantity Required: 150 mL

Container : Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH <3.7. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis. N.B. pH is determined simultaneously.

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA. : 2 standard buffers -4 times daily.

ALKALINITY - GRAN QUALITY CONTROL DATA FROM 03/01/85 TO 24/12/85

Lab: Dorset Analytical Range: 0.24 to 25.00 mg/L as CaCO3

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	268	20.20	20.18	-0.02	0.249
ь	1	270	4.90	4.95	0.05	0.116
a+b		268	25.10	25.13	0.03	0.325
a-b		268	15.30	15.23	-0.07	0.211

s.d.(AB): Sw(within run): 0.149 S(between runs): 0.194 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

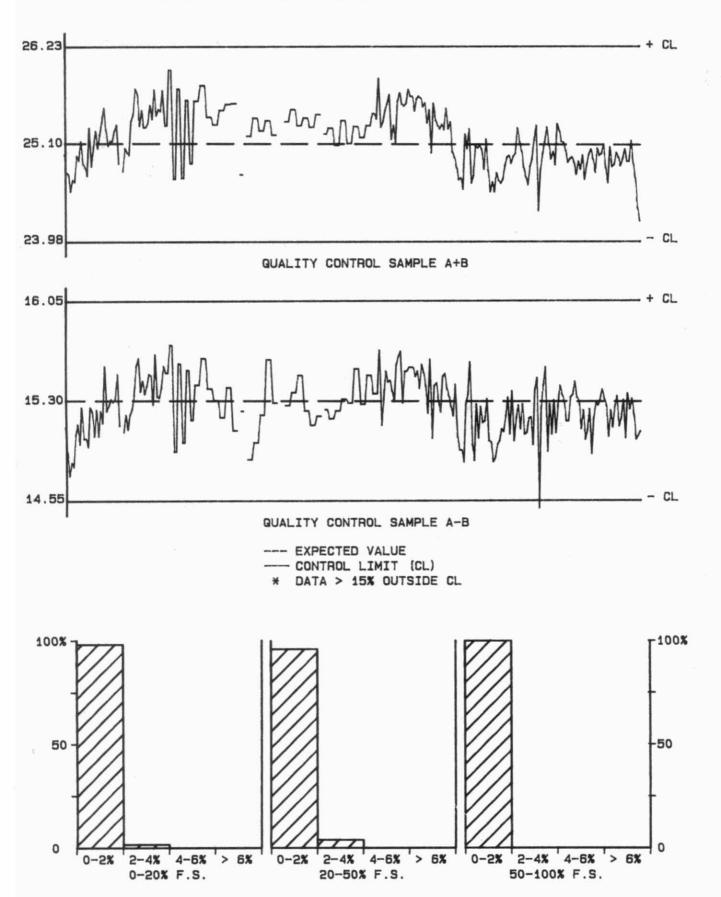
23.97 to 26.22 for A+B 14.55 to 16.05 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	Data Fairs			
	413	0.00 - 2.00	0.078	8.4
	233	2.00 - 5.00	0.121	3.8
	43	5.00 - 10.00	0.146	2.1
	15	10.00 - 25.00	0.155	1.0
	704	Overal1	0.101	N/A

OTHER CHECKS:		Number	Data	Standard(1)	
		of Data	Mean	Deviation	
Long Term Blank	1	258	-0.07	0.099	

QUALITY CONTROL GRAPHS ALKALINITY - GRAN (MG/L AS CACO3)

FROM: 03/01/85 TO: 24/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 25 MG/L AS CACO3

*** ALKALINITY - GRAN ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 09/07/80

LIS Test Name Code: ALKTI Units : mg/L as CaCO3

Work Station Code : RMGALK,RATS Unit Code : 064915
Method Code : 004AT6 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH <4.0. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis.

N.B. pH and total fixed endpoint alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.45*

CALIBRATION:

2 standard buffers covering the pH range 4 to 7.

CONTROLS:

Calibration: LTBL plus two standards, eg, QCA.

Drift: In run standards throughout the run (diluted tap water 20% V/V).

MODIFICATIONS:

02/03/84 -QC program was expanded to include pH and total fixed endpoint alkalinity; preparation and storage of QC solutions was modified.

16/03/84 -Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped liners was recommended for sampling.

09/05/85 -RATS - River Automated Titration System - designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer input (DCI) capabilities.

NOTES:

No data summary available for period not covered in performance report. *Insufficient data available in lowest range to calculate T directly.

ALKALINITY-GRAN QUALITY CONTROL DATA FROM 14/03/85 TO 31/12/85

Lab: Rivers and Lakes

Analytical Range: N/A to 25.00 mg/L as CaCO3

CAL II	BRAT	ION	CONTROL:
--------	------	-----	----------

2 10mm to mar 1 44							
		Number	Expected	Av.Concn	Av.Bias	Standard(1)	
		of Data	of Data Concn Measure			Deviation	
C	:	110	10.00	9.72	-0.28	0.308	
d	1	110	2.50	2.34	-0.16	0.289	
c+d	1	110	12.50	12.05	-0.45	0.530	
c -d	1	110	7.50	7.38	-0.12	0.277	

s.d.(CD): Sw(within run): 0.196 S(between runs): 0.299 S/Sw: 1.52

On any given day the calibration is accepted if the values obtained lie within the ranges:

8.75 to 16.25 for C+D 5.00 to 10.00 for C-D

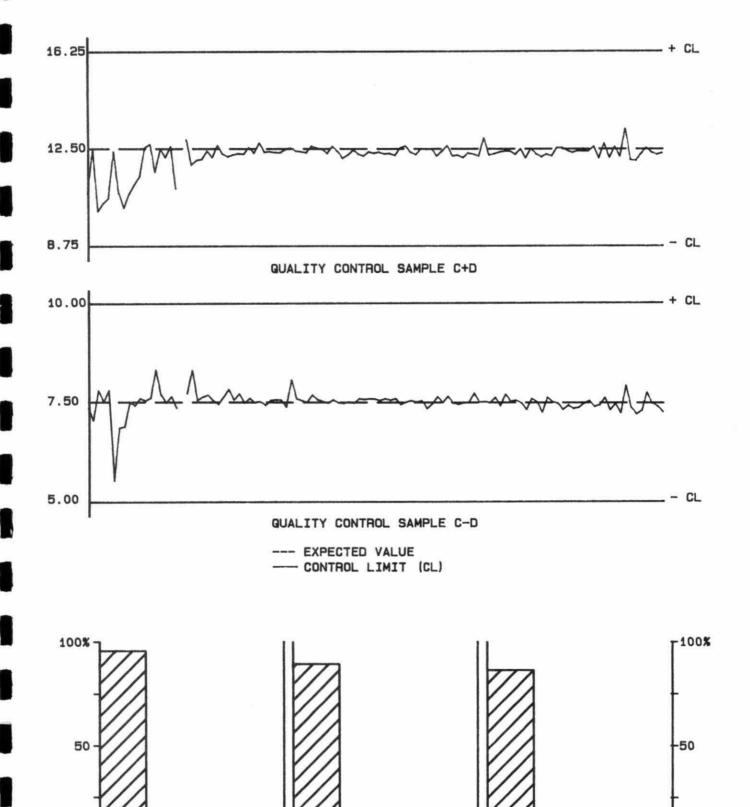
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	2	-2.00 - 0.00	0.135	14.0
	7	0.00 - 2.00	0.149	27.8
	15	2.00 - 5.00	0.203	6.6
	15	5.00 - 10.00	0.865	10.8
	17	10.00 - 25.00	0.706	4.0
	56	Overall	0.605	N/A

DETECTION CRITERION: N/A

OTHER CHECKS:	Nu	ımber	Data	Standard(1)
	of	Data	Me an	Deviation
Long Term Blank	: 19	18	-8.83	0.308

QUALITY CONTROL GRAPHS ALKALINITY-GRAN (MG/L AS CACO3)

FROM: 14/03/85 TO: 31/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 MG/L AS CACO3

2-4% 4-6%

20-50% F.S.

> 6%

0-2%

2-4% 4-6%

50-100% F.S.

0-2%

0-2%

2-4%

0-20% F.S.

4-6%

> 6%

*** ALKALINITY-TOTAL FIXED ENDPOINT (TFE) ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/06/65

LIS Test Name Code: ALKT Units : mg/L as CaCO3

Work Station Code : WALK Unit Code : 064915

Method Code : 003CT3 Supervisor : M. Rawlings Sample Type/Matrix: Domestic Waters, Leachates, Sewage, Industrial Waste,

Effluents

SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples (50.0 mL) are manually pipetted and titrated automatically with 0.02 N sulphuric acid to a pH endpoint of 4.5. Sludges are centrifuged before analysis. INSTRUMENTATION:

- Automated Fisher Titralyzer II system.

REPORTING:

Maximum Significant Figures: 4

Minimum Increment (W): 0.2

Detection Criterion (T): 1.6

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : 1 standard

ALKALINITY-TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 04/01/85 TO 31/12/85

Lab: Domestic Water Analytical Range: 1.6 to 500 mg/L as CaCO3

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	170	285.0	283.7	-1.3	3.32
b	2	170	57.0	59.1	2.1	1.50
a+b		170	342.0	342.8	0.8	4.56
a-b	t	170	228.0	224.6	-3.4	2.40

s.d.(AB): Sw(within run): 1.70 S(between runs): 2.58 S/Sw: 1.52

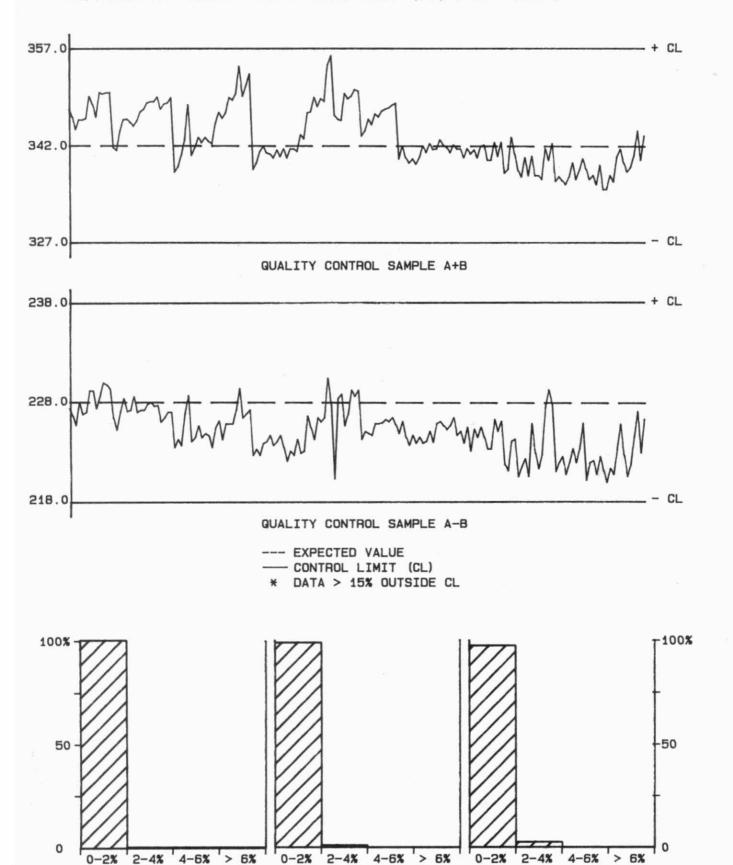
On any given day the calibration is accepted if the values obtained lie within the ranges:

327.0 to 357.0 for A+B 218.0 to 238.0 for A-B

DUPL I CATES	Number of	Sa	mp 1	e	Mean(2)	Coefficient
	Data Pairs	Conc	n S	pan	s.d.	of var.(%)
	68	0.0	-	50.0	0.55	2.3
	82	50.0	-	100.0	0.33	0.4
	110	100	***	200	0.9	0.6
	152	200	-	500	1.8	0.7
	412	Ov	era	11	1.2	N/A

QUALITY CONTROL GRAPHS
ALKALINITY-TOTAL FIXED ENDPOINT (MG/L AS CACO3)

FROM: 04/01/85 TO: 31/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS CACO3

20-50% F.S.

0-20% F.S.

50-100% F.S.

*** ALKALINITY - TOTAL FIXED ENDPOINT (TFE) ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 09/07/80

LIS Test Name Code: ALKT Units : mg/L as CaCO3

Work Station Code : RMGALK, RATS Unit Code : 064915

Method Code : 004AT6 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL

Container : Polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.020 N sulphuric acid to a pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. N.B. pH and Gran alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 4 Minimum Increment (W): 0.01

Detection Criterion (T): 0.78

CALIBRATION:

2 standard buffers covering the pH range 4 to 7.

CONTROLS:

Calibration: LTBL plus 4 standards, eg, QCA.

Drift : In run standards through out the run. Diluted tap water (20% V/V) MODIFICATIONS:

02/03/84- QC program was expanded to include pH and total fixed endpoint alkalinity; preparation and storage of QC solutions was modified.

16/03/84- Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped liners was recommended for sampling.

09/05/85- RATS- River Automated Titration System - designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer (DCI) capabilities.

NOTES:

Control limits were set widely since the performance of the new RATS system was ${\sf unKnown}$.

No data summary is available for the period not covered in performance report.

ALKALINITY-TFE QUALITY CONTROL DATA FROM 03/05/85 TO 31/12/85

Lab: Rivers and Lakes Analytical Range: 0.78 to 250 mg/L as CaCO3

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	97	250.0	249.8	-0.2	1.79
ь	:	97	50.0	50.1	0.1	1.09
a+b	1	97	300.0	299.9	-0.1	2.62
a-b	1	97	200.0	199.7	-0.3	1.40
C	:	93	10.00	9.76	-0.24	0.175
d	1	93	2.50	2.21	-0.29	0.193
c+d		93	12.50	11.97	-0.53	0.277
c -d	:	93	7.50	7.55	0.05	0.242

s.d.(AB): Sw(within run): 0.99 S(between runs): 1.48 S/Sw: 1.50 s.d.(CD): Sw(within run): 0.171 S(between runs): 0.184 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

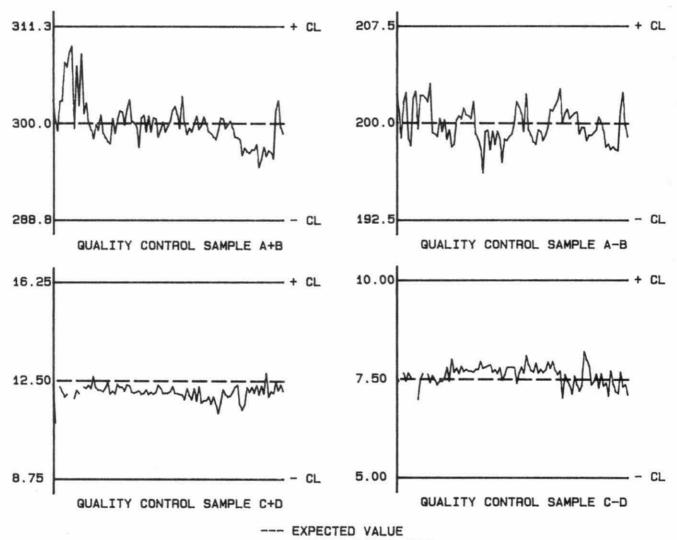
288.8 to 311.2 for A+B 192.5 to 207.5 for A-B 8.75 to 16.25 for C+D 5.00 to 10.00 for C-D

DUPL I CATES :	Number of	San	nple	Mean(2)	Coefficient
	Data Pairs	Concr	Span	s.d.	of var.(%)
	34	0.00 -	5.00	0.261	8.9
	23	5.00	10.00	0.270	3.7
	47	10.00 -	- 25.00	0.510	3.1
	81	25.0	100.0	0.60	0.9
	74	100 -	- 250	0.9	0.5
	259	Ove	erall	0.6	N/A

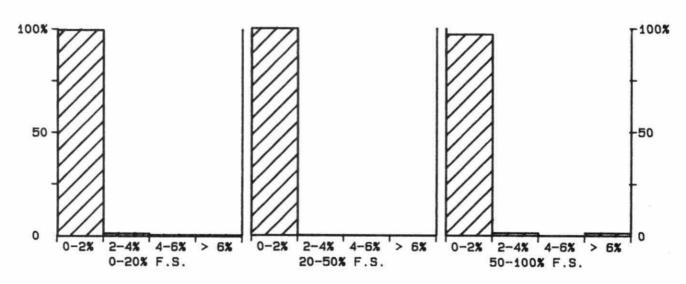
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	ı	94	1.82	0.098

QUALITY CONTROL GRAPHS ALKALINITY-TFE (MG/L AS CACO3)

FROM: 03/05/85 TO: 31/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 250 MG/L AS CACO3

*** CALCIUM ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/06/82
LIS Test Name Code: CAUR Units : mg/L as Ca
Work Station Code : WCAMGH Unit Code : 064820
Method Code : 002AA1 Supervisor : M.Rawlings

Sample Type/Matrix: Domestic Waters, Leachates, Effluents, Sewage

SAMPLING:

Quantity Required: 100 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.16 at the 50 mg/L as Ca level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 0.9

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

MODIFICATIONS:

01/07/82- The method introduced on this date differed slightly from Method B for calcium in HAMES in that full scale for the analytical range was 50.0 mg/L; concentrations for the QC standards were also adjusted.

CALCIUM QUALITY CONTROL DATA FROM 07/01/85 TO 30/12/85

Lab: Domestic Water

Analytical Range: 0.9 to 200 mg/L as Ca

IBRAT		

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	130	130	131	1	1.4
ь	:	130	33	33	0	1.1
a+b	:	130	163	164	1	1.7
a-b	:	130	97	99	2	1.8
c	:	130	32.5	32.7	0.2	0.60
d		130	6.5	6.5	0.0	0.25
c+d	1	130	39.0	39.2	0.2	0.71
c-d	:	130	26.0	26.3	0.3	0.58

s.d.(AB): Sw(within run): 1.3 S(between runs): 1.3 S/Sw: 0.99 s.d.(CD): Sw(within run): 0.41 S(between runs): 0.46 S/Sw: 1.12

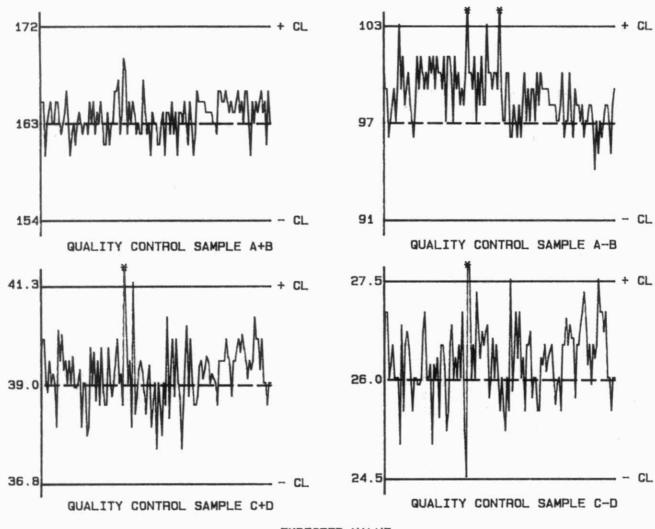
On any given day the calibration is accepted if the values obtained lie within the ranges:

154 to 172 for A+B 91 to 103 for A-B 36.8 to 41.2 for C+D 24.5 to 27.5 for C-D

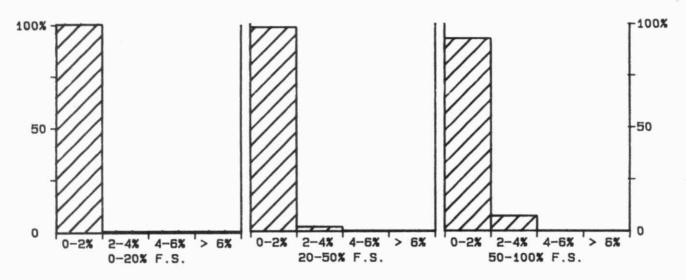
DUPLICATES:	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn	Span	s.d.	of var.(%)
	35	0.0 -	10.0	0.28	7.0
	33	10.0 -	20.0	0.55	3.7
	111	20.0 -	50.0	0.83	2.3
	132	50 -	100	1.2	1.6
	53	100 -	200	1.7	1.3
	364	Over	all	1.1	N/A

QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 07/01/85 TO: 30/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS CA

*** CALCIUM ***

IDENTIFICATION:

: Precipitation Laboratory

Method Introduced: 18/05/79

LIS Test Name Code: CAUR

Units : mg/L as Ca

Work Station Code : PRAA

Unit Code : 064820

Method Code : 002CA1

: M. Raulings Supervisor

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

SAMPLING:

Quantity Required: 5 mL

Container

: Polystyrene

ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 422.7 nm with an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.2 at the 2.00 mg/L level.

INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer(AAS) system

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.08

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Drift

: BL every 10 samples plus 2 standards every 20 samples.

MODIFICATIONS:

17/05/85 - Three additional calibration standards were set up.

- Flow Injection introduction of sample was adopted.
- System was further automated with the addition of a microcomputer to co-ordinate sampler, injection, AAS "read", and data reduction.
- Sample required reduced to 5 mL.

CALCIUM QUALITY CONTROL DATA FROM 08/01/85 TO 20/12/85

Lab: Precipitation Analytical Range: 0.08 to 2.00 mg/L as Ca

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		66	1.20	1.19	-0.01	0.028
ь		67	0.20	0.20	-0.00	0.026
a+b	1	64	1.40	1.39	-0.01	0.041
a-b		64	1.00	0.99	-0.01	0.035

s.d.(AB): Sw(within run): 0.025 S(between runs): 0.027 S/Sw: 1.09

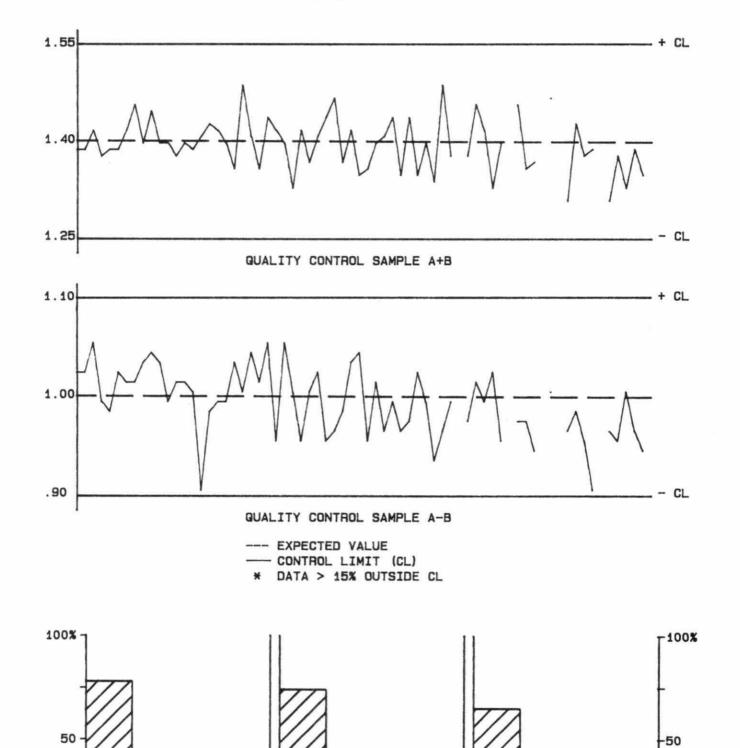
On any given day the calibration is accepted if the values obtained lie within the ranges:

1.25 to 1.55 for A+B 0.90 to 1.10 for A-B

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	56	0.00 - 0.20	0.025	25.4
	54	0.20 - 0.50	0.029	9.0
	21	0.50 - 1.00	0.038	5.3
	18	1.00 - 2.00	0.026	2.0
	149	Overall	0.029	N/A

QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 08/01/85 TO: 20/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS CA

20-50% F.S.

4-6%

> 6%

0-2%

2-4%

50-100% F.S.

4-6%

2-4%

0-2%

0-2%

2-4%

0-20% F.S.

4-6%

*** CALCIUM ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: CAUR Units : mg/L as Ca
Work Station Code : RMAAS Unit Code : 064820
Method Code : 002AA1,002BA1 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: RMAAS: 1.4, full scale value

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W): 0.01 Detection Criterion (T):0.28*,0.33,0.6

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration: LTBL plus 2 standards,eg,QCA for each analytical range Drift: BL plus 1 standard for each analytical range

MODIFICATIONS:

01/12/81- Calibration range became 20.0 mg/L full scale; second analytical range was dropped.

01/03/84- Analytical range(RMCAMGL) was added; full scale: 5.00~mg/L. This range is currently restricted to special programs.

01/09/84- Analytical range(RMCAMGH) was increased from 20.0 to 50.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Magnesium is no longer determined simultaneously. 25/09/85- Calibration range became 35.0 mg/L full scale; second analytical range.

25/09/85- Calibration range became 35.0 mg/L full scale; second analytical range was dropped. Microcomputer controlled system.

NOTES:

Three analytical ranges were used during 1985: 5, 35, and 50 mg/L as Ca full scale. Detection criteria reported above correspond to these ranges respectively.

*T value is based on duplicate analyses at concentrations above the lowest range.

CALCIUM QUALITY CONTROL DATA FROM 03/01/85 TO 09/09/85

Lab: Rivers and Lakes

Analytical Range: 0.6 to 50.0 mg/L as Ca

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	40	37.5	37.8	0.3	0.61
ь		40	12.5	12.7	0.2	0.21
a+b	1	40	50.0	50.5	0.5	0.67
a-b		40	25.0	25.1	0.1	0.63

s.d.(AB): Sw(within run): 0.45 S(between runs): 0.46 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

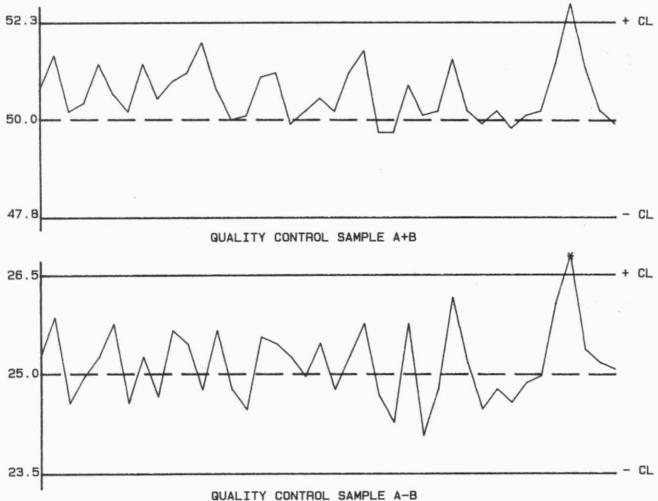
47.8 to 52.2 for A+B 23.5 to 26.5 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	9	0.0 - 2.0	0.20	15.7
	38	2.0 - 5.0	0.36	12.1
	11	5.0 - 10.0	0.29	4.2
	17	10.0 - 20.0	0.46	3.2
	20	20.0 - 50.0	0.57	1.7
	95	Overal1	0.41	N/A

OTHER CHECKS:		Number	Data	Standard(1)	
	of Data		Mean	Deviation	
Absorbance	:	30	1.08	0.134	
Long Term Blank	:	0	N/A	N/A	

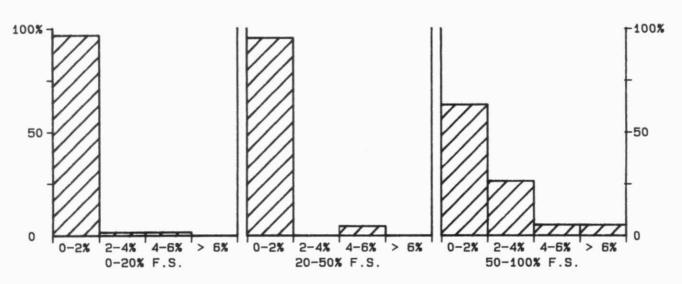
QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 03/01/85 TO: 09/09/85



GUALITY CONTROL SAMPLE

--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS CA

CALCIUM QUALITY CONTROL DATA FROM 26/09/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.33 to 35.00 mg/L as Ca

CAL.	BRAT	ION	CON	TROL:
UI IL		1011	COIT	I I COL .

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	28	28.00	28.09	0.09	0.325
ь	1	28	2.45	2.49	0.04	0.057
a+b	1	28	30.45	30.58	0.13	0.363
a-b	1	28	25.55	25.60	0.05	0.294

s.d.(AB): Sw(within run): 0.208 S(between runs): 0.233 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

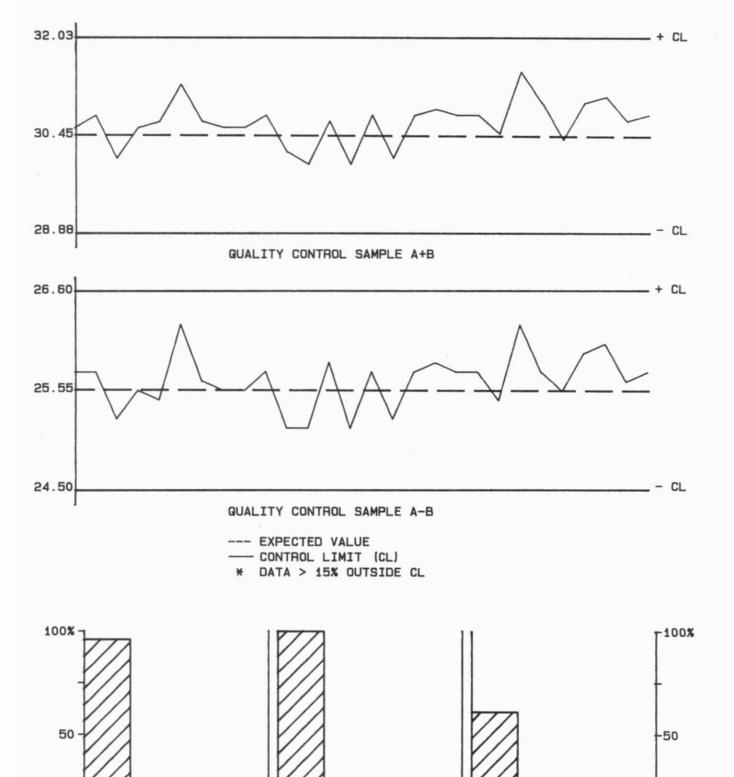
28.87 to 32.02 for A+B 24.50 to 26.60 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	7	0.00 - 1.75	0.110	6.7
	46	1.75 - 3.50	0.165	6.5
	10	3.50 - 7.00	0.315	6.8
	14	7.00 - 17.50	0.333	3.2
	1	17.50 - 35.00	N/A	N/A
	78	Overall	0.228	N/A

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	Ø	N/A	N/A
Long Term Blank	:	Ø	N/A	N/A

QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 25/09/85 TO: 30/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 35 MG/L AS CA

2-4% 4-6%

20-50% F.S.

> 6%

0-2%

2-4%

4-6%

50-100% F.S.

0-2%

2-4% 4-6%

0-20% F.S.

0-2%

CALCIUM QUALITY CONTROL DATA FROM 07/01/85 TO 10/09/85

Lab: Rivers and Lakes

Analytical Range: N/A to 5.00 mg/L as Ca

CALIBRATION CONTROL	CAL	CONTROL	ᇿ
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		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	49	3.75	3.76	0.01	0.057
ь		49	1.25	1.24	-0.01	0.035
a+b	:	49	5.00	5.01	0.01	0.076
a-b		49	2.50	2.52	0.02	0.057

s.d.(AB): Sw(within run): 0.040 S(between runs): 0.047 S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 4.77 to 5.23 for A+B 2.35 to 2.65 for A-B

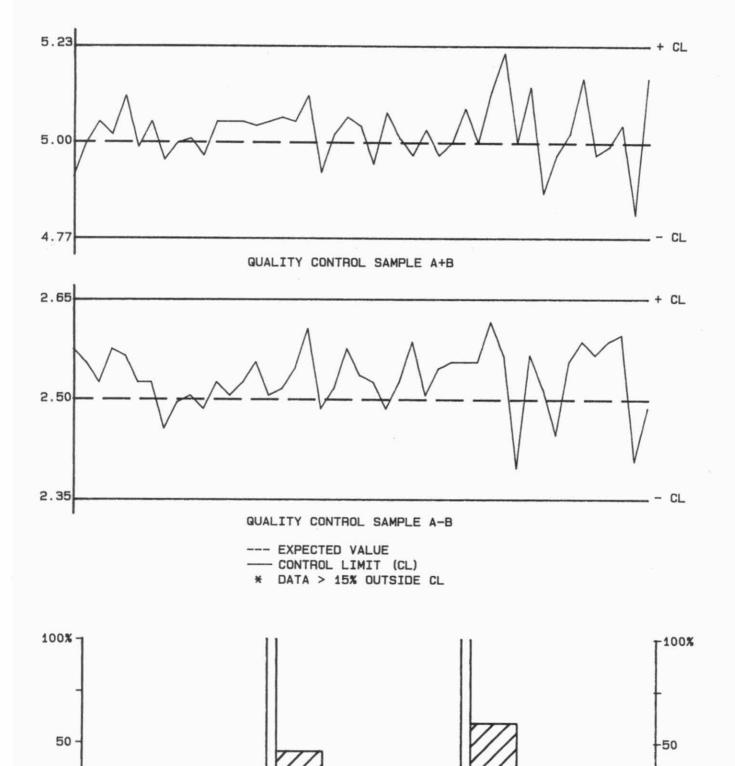
DUPL ICATES:	Number of	Sample	:	Mean(2)	Coefficient
	Data Pairs	Concn Sp	an	s.d.	of var.(%)
	Ø	0.00 -	1.00	N/A	N/A
	15	1.00 -	2.00	0.094	5.5
	65	2.00 -	3.00	0.116	4.7
	49	3.00 -	5.00	0.090	2.4
	129	Overal	1	0.105	N/A

DETECTION CRITERION: N/A

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	42	0.72	0.414
Long Term Blank	:	Ø	N/A	N/A

QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 07/01/85 TO: 10/09/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS CA

20-50% F.S.

2-4%

4-6%

2-4%

4-6%

50-100% F.S.

0

0-2%

2-4% 4-6%

0-20% F.S.

> 6%

0-2%

*** CARBON - DISSOLVED INORGANIC ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 03/06/80 Supervisor : F. Tomassini Units : mg/L as C

Sample Type/Matrix: Streams, Lakes

SAMPLING:

Quantity Required: 50 mL

Container : Pyrex culture tubes plus screw caps with cone-shaped liners

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the 10 mg/L level.

INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: air (CO2-free) supply, 60 C heating bath(7.7 mL delay), dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.05

CALIBRATION:

BL plus 9 standards daily.

CONTROLS

Calibration: LTBL plus 4 standards,eg,QCA

Drift : BL every 10 samples and BL plus 1 standard every 20 samples.

NOTES:

As concentration of calibration control solutions slowly change with time at these low concentrations, calibration control ranges are based on measured averages rather than expected concentrations.

CARBON - DISSOLVED INORGANIC QUALITY CONTROL DATA FROM 08/01/85 TO 20/12/85

Lab: Dorset

Analytical Range: 0.05 to 10.00 mg/L as C

COLI	DDAT	TON	COAR	mon .
CHLI	DKHI	TOM	CUIT	TROL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a.	. 2	152	6.4	6.6	0.2	0.16
ь	=	152	2.2	2.3	0.1	0.09
a+b		152	8.6	8.8	0.2	0.22
a-b	1	152	4.2	4.3	0.1	0.15
c		151	1.39	1.47	0.08	0.042
d		151	0.63	0.61	-0.02	0.039
c+d		151	2.02	2.08	0.06	0.073
c-d		151	0.76	0.86	0.10	0.035

s.d.(AB): Sw(within run): 0.11 S(between runs): 0.13 S/Sw: 1.22 s.d.(CD): Sw(within run): 0.025 S(between runs): 0.041 S/Sw: 1.64

On any given day the calibration is accepted if the values obtained lie within the ranges:

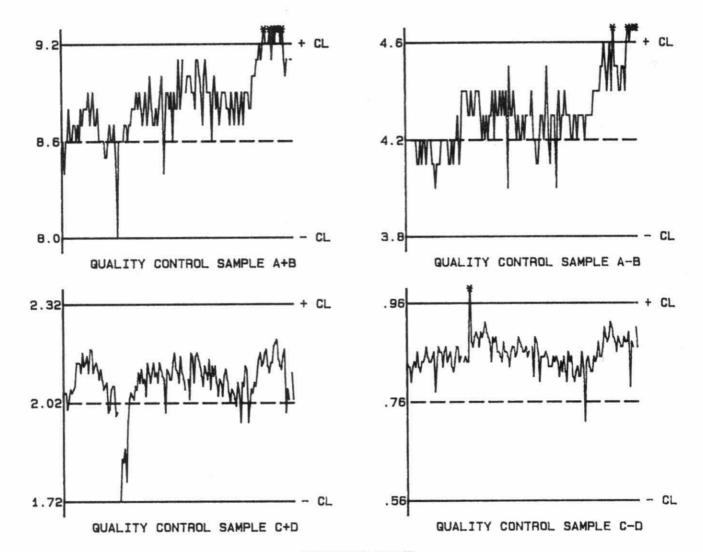
8.0 to 9.2 for A+B 3.8 to 4.6 for A-B 1.72 to 2.32 for C+D 0.56 to 0.96 for C-D

DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	92	0.00 - 0.50	0.017	4.9
	122	0.50 - 1.00	0.018	2.2
	153	1.00 - 2.00	0.033	2.3
	65	2.00 - 5.00	0.075	2.6
	17	5.00 - 10.00	0.177	2.6
	449	Overall	0.050	N/A

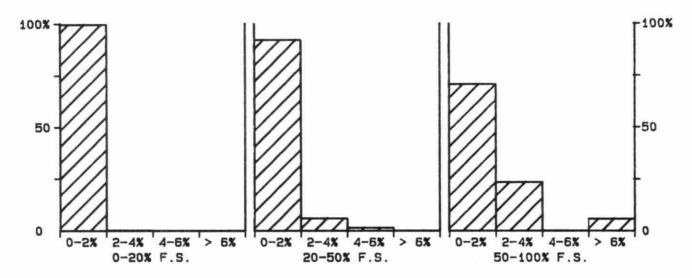
OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Me a.n	Deviation
STD. CAL.	1	148	565	48.3
Long Term Blank	:	151	0.18	0.029

QUALITY CONTROL GRAPHS CARBON - DISSOLVED INORGANIC (MG/L AS C)

FROM: 08/01/85 TO: 20/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS C

*** CARBON - DISSOLVED INORGANIC ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78

LIS Test Name Code: DIC Units : mg/L as C Work Station Code: ROC Unit Code : 064806

Method Code : 102AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.4 at the 40 mg/L level.

N.B. Dissolved organic carbon is determined simultaneously.

INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: air (CO2-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.2

Detection Criterion (T): 0.5

CALIBRATION:

BL plus 1 standard daily. Bl plus 4 standards whenever a new stock of buffer is prepared.

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard

CARBON - DISSOLVED INORGANIC QUALITY CONTROL DATA FROM 04/01/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.46 to 40.00 mg/L as C

CALIBRATION CONTROL:

C. 1 100 0 00 1 /1						
		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	155	30.00	30.18	0.18	0.332
b	:	155	10.00	10.09	0.09	0.287
a+b	:	155	40.00	40.27	0.27	0.510
a-b	1	155	20.00	20.09	0.09	0.354

s.d.(AB): Sw(within run): 0.250 S(between runs): 0.310 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

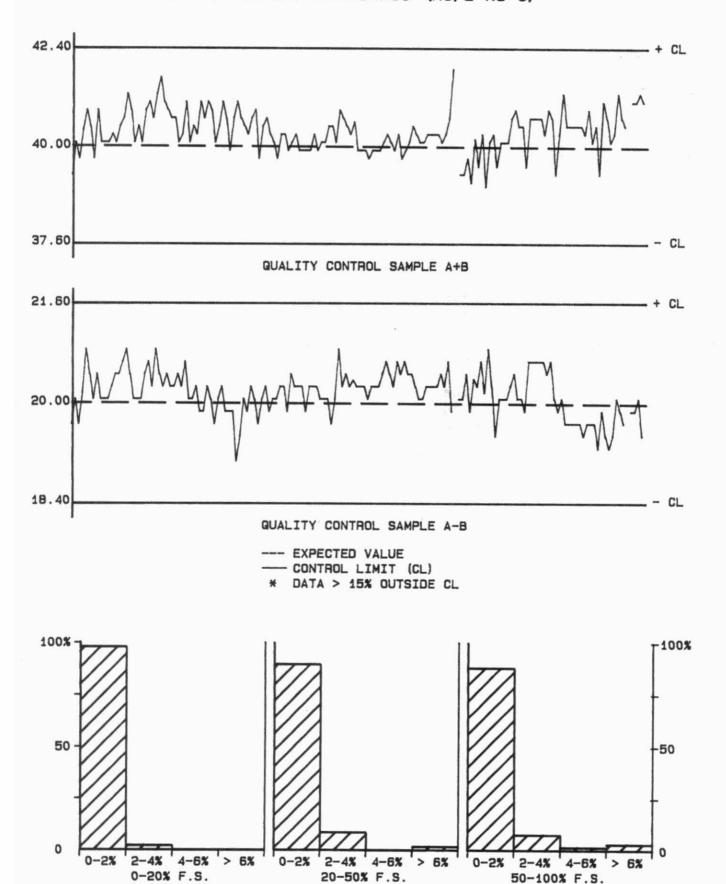
37.60 to 42.40 for A+B 18.40 to 21.60 for A-B

DUPL I CATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	167	0.00 - 4.00	0.154	13.4
	38	4.00 - 10.00	0.473	6.9
	47	10.00 - 20.00	0.211	1.4
	66	20.00 - 40.00	0.565	2.1
	318	Overall	0.335	N/A

OTHER CHECKS:	Number of Data		Data Mean	Standard(1) Deviation
Std Cal	:	141	393	100.9
Long Term Blank	:	152	0.1	0.11

QUALITY CONTROL GRAPHS CARBON - DISSOLVED INORGANIC (MG/L AS C)

FROM: 04/01/85 TO: 13/02/88



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 MG/L AS C

*** CARBON - DISSOLVED ORGANIC ***

IDENTIFICATION:

Laboratory | Rivers and Lakes | Method Introduced: 01/04/78

LIS Test Name Code: DOC Units : mg/L as C Work Station Code: ROC Unit Code : 064806

Method Code : 102AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Waters, Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas(500 mL/min) to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light(UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the 20 mg/L level.

N.B. Dissolved inorganic carbon is determined simultaneously.

INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO2-free) gas supplies with flow controls, dialysis unit, UV digester. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 0.3

CALIBRATION

BL plus 1 standard daily. BL plus 4 standards whenever a new stock of buffer is prepared.

CONTROLS:

Calibration : LTBL plus 2 standards,eg,QCA

Drift | BL plus 1 standard

CARBON - DISSOLVED ORGANIC QUALITY CONTROL DATA FROM 04/01/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.28 to 20.00 mg/L as C

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		157	15.00	15.14	0.14	0.167
ь	ı	157	5.00	5.00	0.00	0.094
a+b	:	157	20.00	20.15	0.15	0.205
a-b	:	157	10.00	10.14	0.14	0.177

s.d.(AB): Sw(within run): 0.125 S(between runs):

0.136 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

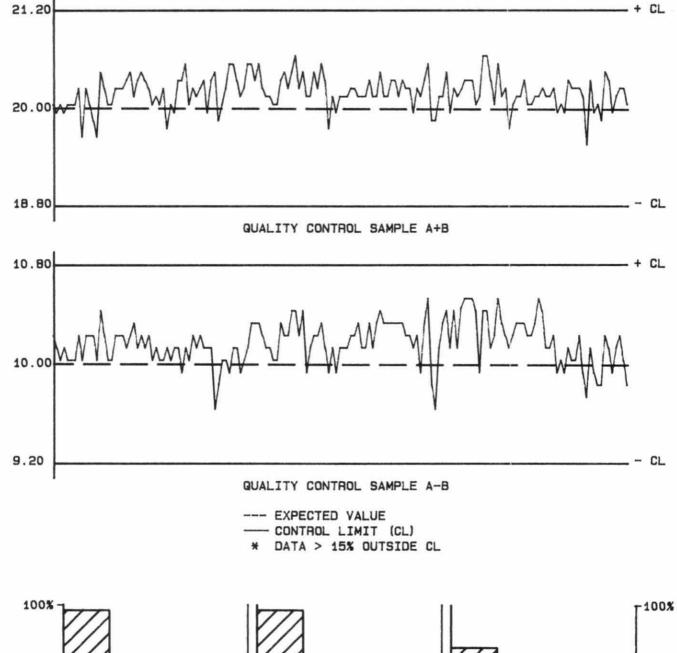
18.80 to 21.20 for A+B 9.20 to 10.80 for A-B

DUPL I CATES :	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	118	0.00 - 2.00	0.095	8.8
	189	2.00 - 5.00	0.111	3.3
	76	5.00 - 10.00	0.131	1.9
	29	10.00 - 20.00	0.242	1.8
	412	Overal1	0.124	N/A

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Std Cal		144	477	121.1
Long Term Blank		155	0.1	0.05

QUALITY CONTROL GRAPHS CARBON - DISSOLVED ORGANIC (MG/L AS C)

FROM: 04/01/85 TO: 30/12/85



100% 50 0 0-2% 2-4% 4-6% > 6% 0-2% 2-4% 4-6% > 6% 0-2% 2-4% 4-6% > 6% 0-20% F.S. 50-100% F.S.

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS C

*** CHLORIDE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/06/66

LIS Test Name Code: CLIDUR Units : mg/L as Cl

Work Station Code : WCL Unit Code : 064817

Method Code : 002BT3 Supervisor : M. Rawlings Sample Type/Matrix: Domestic Waters, Leachates, Sewage, Industrial Waste,

Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

An aliquot(10.0 mL) of sample is automatically pipetted and titrated with silver nitrate to an endpoint which is determined by calibration. The addition of silver nitrate to a sample containing chloride ions results in the precipitation of silver chloride with a corresponding change in voltage between the electrodes. The endpoint is equivalent to the maximum change in voltage per unit volume of titrant. A reagent containing acetone and nitric acid is utilized to prevent fouling of the electrodes by silver chloride precipitate.

INSTRUMENTION:

Radiometer ATS-1 autopipetting titration system equipped with a silver-silver chloride electrode and a non-calomel reference electrode.

REPORTING:

Maximum Significant Figures: 4
Minimum Increment (W): 0.2

Detection Criterion (T): 1.3

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : Acid wash, BL, plus 1 standard

CHLORIDE QUALITY CONTROL DATA FROM 02/01/85 TO 27/12/85

Lab: Domestic Water

Analytical Range: 1.3 to 500.0 mg/L as Cl

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	112	170.0	169.6	-0.4	1.67
b		112	17.0	17.1	0.1	0.52
a+b		112	187.0	186.7	-0.3	1.73
a-b		112	153.0	152.5	-0.5	1.78

s.d.(AB): Sw(within run): 1.26 S(between runs): 1.24 S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

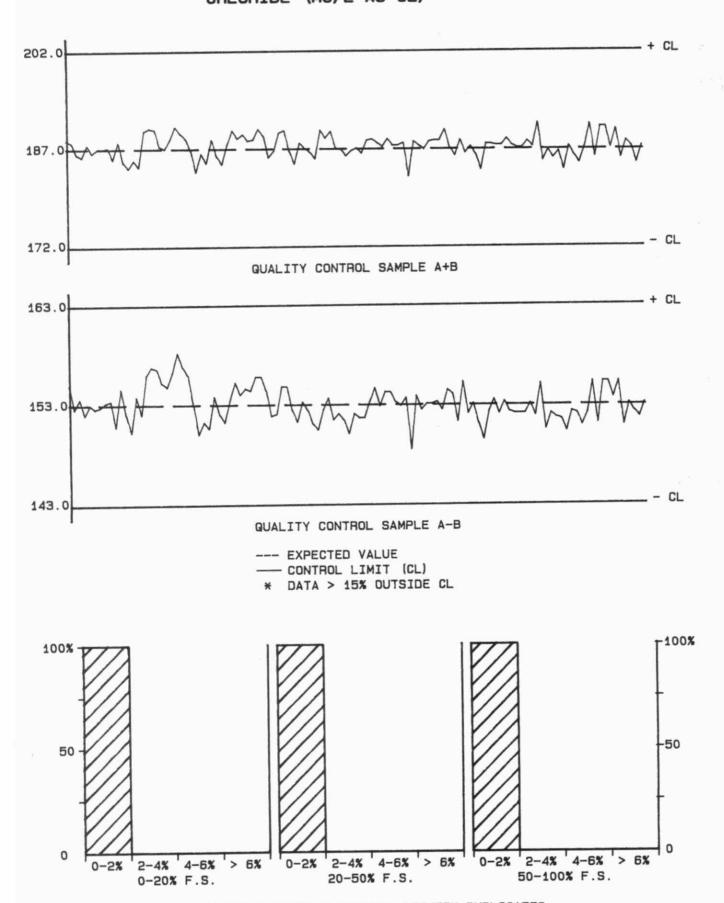
> 172.0 to 202.0 for A+B 143.0 to 163.0 for A-B

DUPL I CATES :	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	198	0.0 - 50.0	0.44	2.7
	45	50.0 - 100.0	0.88	1.2
	23	100.0 - 250.0	1.12	0.7
	8	250.0 - 500.0	2.57	0.8
	274	Overall	0.75	N/A

DETECTION CRITERION: 1.3

QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 02/01/85 TO: 27/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS CL

*** CHLORIDE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/04/78
LIS Test Name Code: CLIDUR Units : mg/L as Cl
Work Station Code : PRIC1 Unit Code : 064817
Method Code : 005AI0 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required: 15 ml.

Container : Polystyrene bottle.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample scan to a series of standard scans.

Full scale conductivity : 10 uS/cm.

Nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.05

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards (eg) QCA

Drift : 1 standard every 10 samples.

MODIFICATIONS:

20/09/84 - Chloride range was changed from 1.50 mg/L full scale to 2.00 mg/L full scale.

12/04/85 - Chloride quality control standards were changed; QCA from 1.20 to 1.60 mg/L, QCB from 0.30 to 0.40 mg/L. First three months' data were omitted because they were not comparable with the later ones.

CHLORIDE QUALITY CONTROL DATA FROM 12/04/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 0.05 to 2.00 mg/L as Cl

CALIBRATION CONTROL:	CAL	IBRAT	HOI	CONTRO	LI
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		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	1	110	1.60	1.59	-0.01	0.012
b	1	111	0.40	0.40	-0.00	0.016
a+b	:	110	2.00	1.99	-0.01	0.020
a-b		110	1.20	1.20	-0.00	0.019

s.d.(AB): Sw(within run): 0.013 S(between runs): 0.014 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

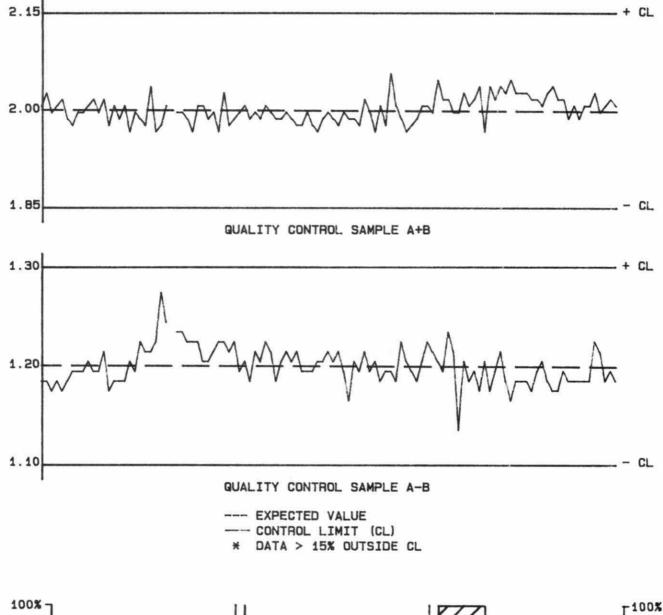
1.85 to 2.15 for A+B 1.10 to 1.30 for A-B

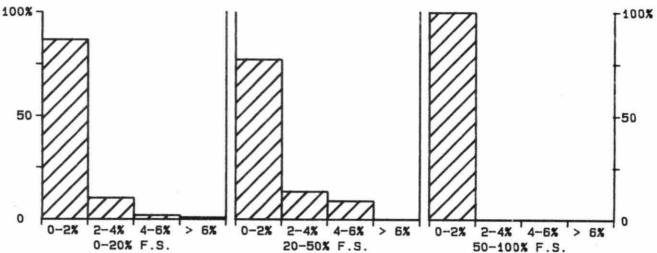
DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	57	0.00 - 0.20	0.018	16.5
	56	0.20 - 0.50	0.028	8.9
	15	0.50 - 1.00	0.027	4.1
	2	1.00 - 2.00	0.010	0.6
	130	Overal1	0.024	N/A

DETECTION CRITERION: 0.05

QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 12/04/85 TO: 23/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 MG/L AS CL

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/04/78

LIS Test Name Code: CLIDUR Units : ug/Filter as Cl

Work Station Code : PRLOV Unit Code : 361817
Method Code : 004AIC Supervisor : M. Rawlings

Sample Type/Matrix: W40 filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bag

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as Cl.

Full scale conductivity : 30 uS/cm.

Nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Ultrasonic bath; polyethylene tubes

Automated modular continuous flow ion chromatographic system.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment(W): 0.50 ug/filter Detection Criterion(T): 0.8 ug/filter

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

MODIFICATIONS:

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

20/09/84 - Chloride range was changed from 1.50 mg/L full scale to 2.00 mg/L full scale. Quality control standards were not changed.

12/04/85 - Chloride quality control standards were changed; QCA from 1.20 to 1.60 mg/L and QCB from 0.30 to 0.40 mg/L. First three months' data were omitted because they were not comparable with the later ones.

10/05/85 - Microcomputer used for data reduction. Three additional calibration standards were set up.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

CHLORIDE QUALITY CONTROL DATA FROM 30/04/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 0.8 to 100.0 ug/filter as Cl

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	25	80.0	79.4	-0.6	1.03
ь	1	25	20.0	20.4	0.4	0.56
a+b	1	25	100.0	99.8	-0.2	1.27
a-b		25	60.0	59.0	-1.0	1.08

s.d.(AB): Sw(within run): 0.76 S(between runs):

0.83 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

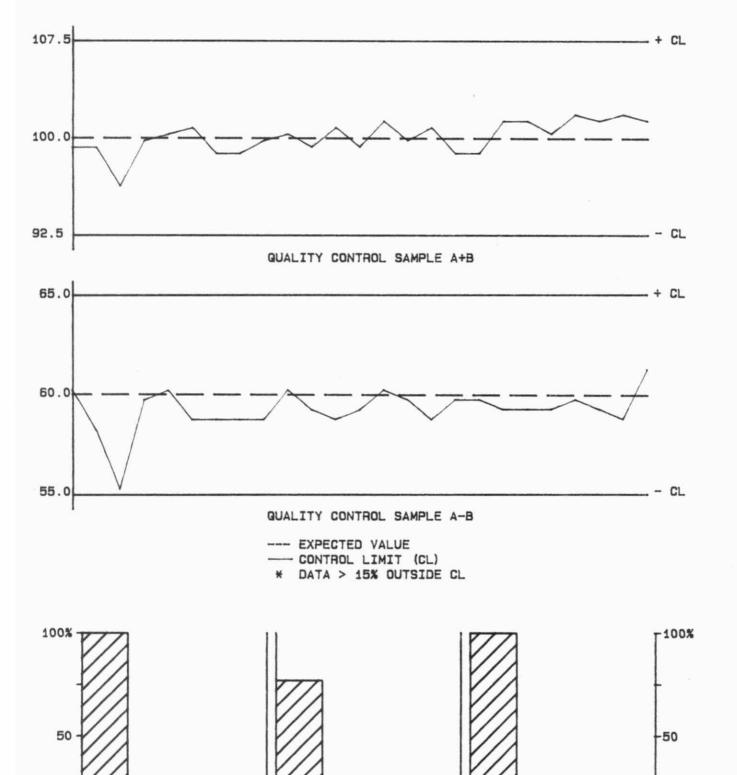
> 92.5 to 107.5 for A+B 55.0 to 65.0 for A-B

DUPLICATES:	Number of Sample		Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	8	0.0 - 15.0	0.28	9.2
	10	15.0 - 37.5	0.62	2.7
	9	37.5 - 100.0	1.29	2.7
	27	Overall	0.85	N/A

DETECTION CRITERION: 0.8

QUALITY CONTROL GRAPHS CHLORIDE (UG/FILTER AS CL)

FROM: 30/04/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 UG/FILTER AS CL

20-50% F.S.

4-6%

> 6%

0-2%

2-4% 4-6% > 6%

50-100% F.S.

2-4%

0-2%

2-4% 4-6% > 6%

0-20% F.S.

*** CHLORIDE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/05/75
LIS Test Name Code: CLIDUR Units : mg/L as Cl

Work Station Code : RMSICL Unit Code : 064817
Method Code : 004AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts. Effluents.

SAMPLING:

Quantity Required: 50 ml

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate(red), and the absorbance of the latter is measured colourimetrically. A reference stream, from which mercuric thiocyanate has been eliminated, is utilized tocompensate for sample matrix effects. Approximate absorbance: 0.3 at the 50 mg/L level N.B. Reactive silicates are determined simultaneously.

INSTRUMENTATION:

Boxed-FIA system consisting of basic automated modular continuous flow system plus the following modules: sample injection valves with air-flow controls, timer, bubble-gate. Colourimetric measurement is through a 1.5 cm light path at 470nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.24

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration: LTBL plus 3 standards, eg,QCA

Drift : BL plus 4 standard

MODIFICATIONS:

04/07/83 - Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10, and concentrations of QC standards were adjusted. The analytical rate was tripled.

CHLORIDE QUALITY CONTROL DATA FROM 03/01/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.24 to 50.0 mg/L as Cl

CAL	IBRAT	HOLL	CONTROL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a.	:	66	30.0	30.0	0.0	0.41
b		66	8.0	8.1	0.1	0.17
a+b	:	66	38.0	38.1	0.1	0.50
a-b	:	66	22.0	21.9	-0.1	0.38
c	:	66	8.00	8.05	0.05	0.115
d	:	66	2.00	2.04	0.04	0.060
c+d		66	10.00	10.09	0.08	0.133
c-d		66	6.00	6.00	0.00	0.126

s.d.(AB): Sw(within run): 0.27 S(between runs): 0.31 S/Sw: 1.17 s.d.(CD): Sw(within run): 0.089 S(between runs): 0.092 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

35.8 to 40.2 for A+B 20.5 to 23.5 for A-B 9.55 to 10.45 for C+D 5.70 to 6.30 for C-D

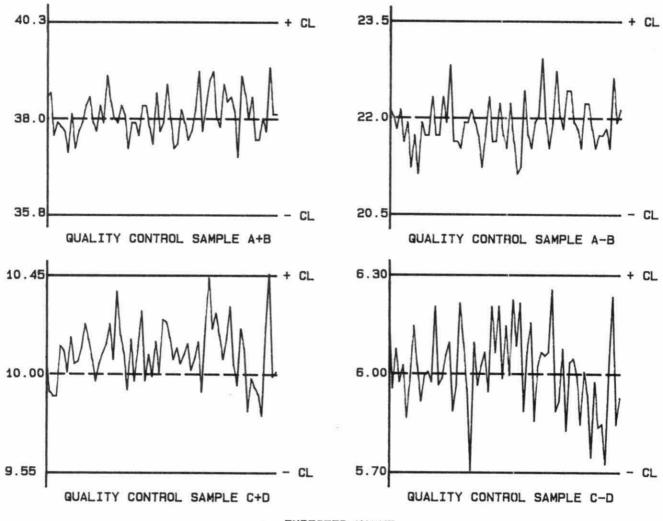
DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	116	0.00 - 2.00	0.081	14.3
	16	2.00 - 5.00	0.233	6.9
	22	5.00 - 10.00	0.162	2.2
	61	10.0 - 25.0	0.35	1.8
	24	25.0 - 50.0	0.46	1.4
	239	Overal1	0.25	N/A

DETECTION CRITERION: 0.24

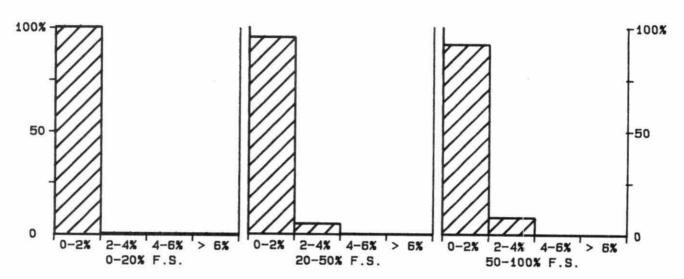
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal.	:	66	543	23.3
Long Term Blank	:	66	0.00	0.002

QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 03/01/85 TO: 30/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS CL

*** CHLOROPHYLL ***

IDENTIFICATION:

Method Introduced: 01/04/75 Laboratory Rivers and Lakes LIS Test Name Code: CHLRAT, CHLRBT, CHLRAC Units : ug/L Work Station Code : RCHLO Unit Code : 063000

Method Code : 002DS1 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPLING:

Quantity Required: 1000 mL : Glass Container

Other : In the field a sample is filtered through a 1.2 u cellulose nitrate membrane filter. The filter is then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish.

SAMPLE PREPARATION:

If the sample has not been filtered in the field, a measured volume is filtered through a 1.2 um cellulose nitrate membrane filter under moderate suction. After the addition of 4.5 mL acetone (90% V/V), the filter is ground to release chlorophyll using glass beads; an additional 4.5 mL acetone (90% V/V) is added, and the mixture is rested overnight to improve extraction efficiency. After filtration through Whatman 934AH glass fibre filters (no suction), the volume is adjusted to 12.0 mL followed by centrifugation.

ANALYTICAL PROCEDURE:

Using a microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 665 nm; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll a and b are calculated from the first scan. After automated acidification, the second scan is obtained for the same wavelengths, and used for calculating chlorophyll a, corrected. SCOR-UNESCO equations are used for all chlorophyll calculations.

INSTRUMENTION:

-Vacuum filtration modules; centrifuge set at 2500 rpm. Automated modular continuous flow scanning spectrophotometer system. -Microcomputer system for control of sampling, timing, and data processing (i.e. data capture, calculations, and transfer of results to LIS).

REPORTING

Maximum Significant Figures: 3 Minimum Increment (W) : 0.01

Detection Criterion (T): 0.51,0.31,0.43

CONTROLS

Calibration : LTBL plus 2 "standards", eg, QCA

Drift : 1 "standard"

MODIFICATIONS:

01/07/84 -Automated, microcomputer controlled system was introduced. 13/06/85 -Centrifuging steps were elimininated and nylon filters were introduced.

NOTES:

In 1982, calibration controls were stable, but were prepared from dyes rather than chlorophyll. "Standards" are now prepared from chlorophyll a and b, but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages. No data summary is available for period not covered in performance report.

CHLOROPHYLL - a QUALITY CONTROL DATA FROM 13/06/85 TO 17/12/85

Lab: Rivers and Lakes

Analytical Range: 0.51 to 10.00 ug/L

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	83	3.06	2.96	-0.10	0.094
b	:	83	1.02	1.00	-0.02	0.056
a+b	:	83	4.08	3.96	-0.12	0.139
a-b	:	83	2.04	1.95	-0.09	0.068

s.d.(AB): Sw(within run): 0.048 S(between runs): 0.077 S/Sw: 1.61

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.58 to 5.58 for A+B 1.04 to 3.04 for A-B

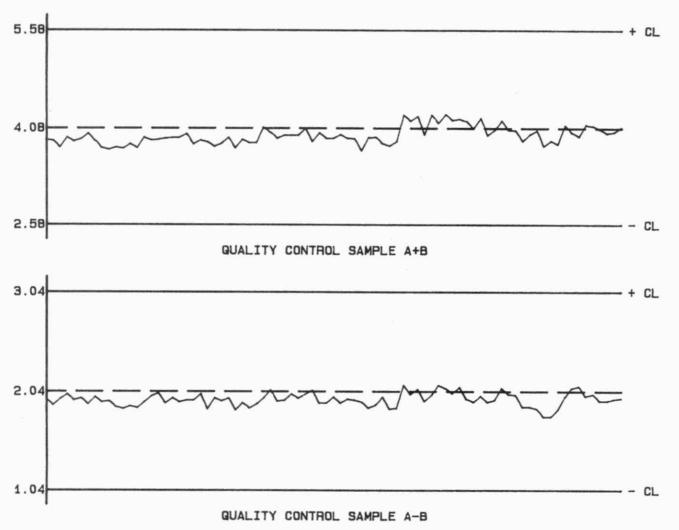
DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	37	0.00 - 2.00	0.171	13.3
	24	2.00 - 5.00	0.298	9.7
	10	5.00 - 10.00	1.067	14.5
	71	Overal1	0.453	N/A

DETECTION CRITERION: 0.51

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Long Term Blank	:	83	0.05	0.032
Digested Blank	:	83	0.06	0.036

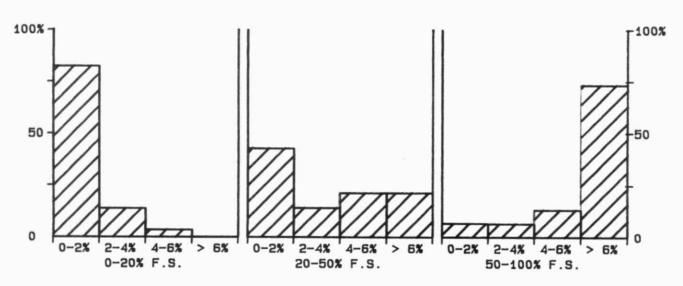
QUALITY CONTROL GRAPHS CHLOROPHYLL - A (UG/L)

FROM: 13/06/85 TO: 17/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)

* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 UG/L

CHLOROPHYLL - b QUALITY CONTROL DATA FROM 13/06/85 TO 18/12/85

Lab: Rivers and Lakes

Analytical Range: 0.31 to 10.00 ug/L

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	85	3.06	2.99	-0.07	0.137
ь	:	85	1.02	1.03	0.01	0.075
a+b	:	85	4.08	4.02	-0.06	0.200
a-b	:	85	2.04	1.96	-0.08	0.094

s.d.(AB): Sw(within run): 0.066 S(between runs): 0.110 S/Sw: 1.66

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.33 to 4.83 for A+B 1.54 to 2.54 for A-B

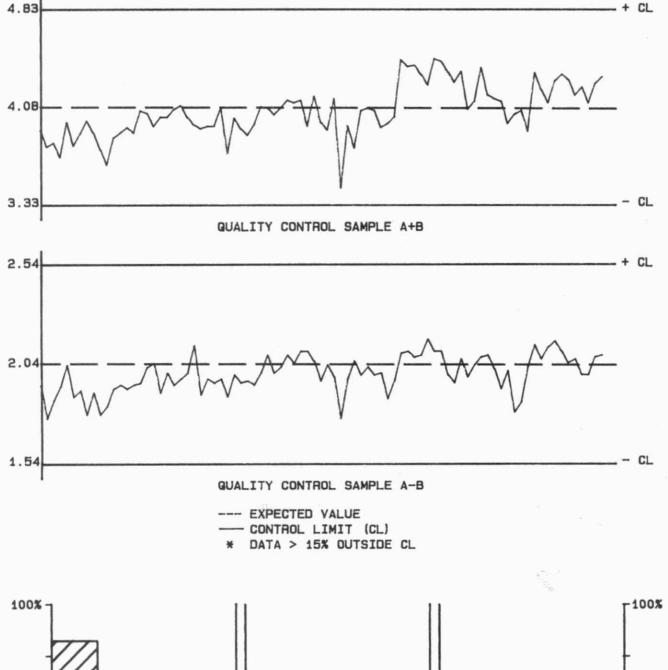
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	74	0.00 - 2.00	0.102	15.9
	5	2.00 - 5.00	0.462	13.9
	2	5.00 - 10.00	0.328	3.7
	81	Overall	0.159	N/A

DETECTION CRITERION: 0.31

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	84	0.06	0.046
Digested Blank	:	84	0.07	0.055

QUALITY CONTROL GRAPHS CHLOROPHYLL - B (UG/L)

FROM: 13/06/85 TO: 18/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 10 UG/L

CHLOROPHYLL-ACIDIFIED QUALITY CONTROL DATA FROM 14/06/85 TO 17/12/85

Lab: Rivers and Lakes

Analytical Range: 0.43 to 10.00 ug/L

COL	TDDAT	THE	CON	TROL:
	IDRO	1014	CUIT	I INDL.

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	65	2.50	2.50	0.00	0.212
b	1	65	1.00	0.80	-0.20	0.129
a+b	:	65	3.50	3.30	-0.20	0.304
a-b	:	65	1.50	1.70	0.20	0.176

s.d.(AB): Sw(within run): 0.124 S(between runs): 0.175 S/Sw: 1.41

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.00 to 5.00 for A+B 0.50 to 2.50 for A-B

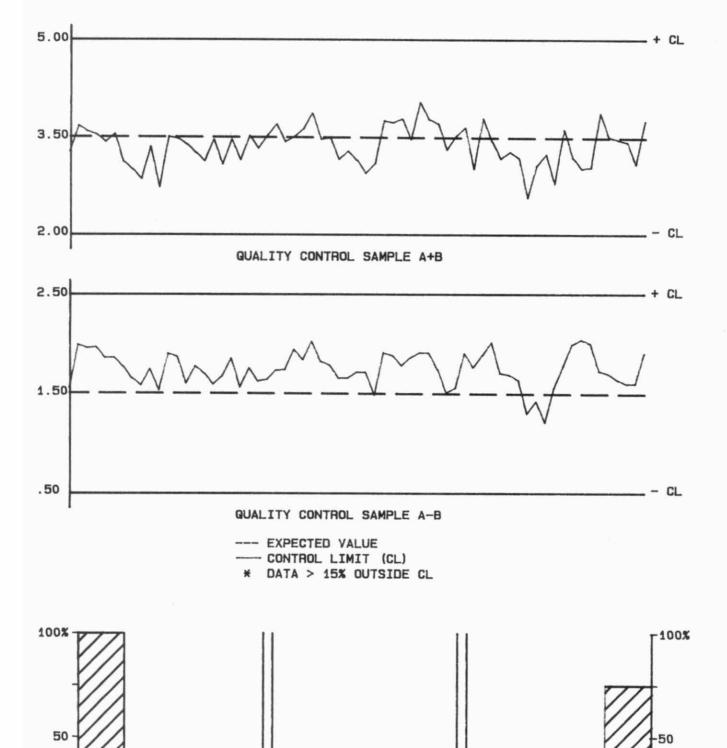
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	9	0.00 - 3.00	0.144	15.3
	2	3.00 - 5.00	0.235	5.8
	2	5.00 - 10.00	0.855	13.6
	13	Overall	0.368	N/A

DETECTION CRITERION: 0.43

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	65	0.04	0.086
Digested Blank		65	0.03	0.094

QUALITY CONTROL GRAPHS CHLOROPHYLL-ACIDIFIED (UG/L)

FROM: 14/06/85 TO: 17/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 20 UG/L

20-50% F.S.

4-6%

> 6%

0-2%

2-4%

50-100% F.S.

2-4%

0-2%

2-4% 4-6%

0-20% F.S.

> 6%

0-2%

*** COLOUR - TRUE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 13/03/84

LIS Test Name Code: COLTR Units : TCU
Work Station Code: WCOL Unit Code : 342000
Method Code : 102BC9 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.05 at the 70 TCU level.

INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter(400-450nm). Turbitly measurement is through a 5.0 cm. light path using a different broadband filter(660-740nm).

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.5

Detection Criterion (T): 1.1

CALIBRATION:

BL plus 1 standard in duplicate

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard

NOTES:

New procedure was initiated to conform with change in "Ontario Drinking Water Objectives"; copy of research study is available on request.

COLOUR-TRUE QUALITY CONTROL DATA FROM 03/01/85 TO 31/12/85

Lab: Domestic Water

Analytical Range: 1.1 to 100.0 TCU

CAL	TAGG	HOLL	CONTROL	٠
CITE	DIVITI	TOLA	CONTROL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	64	50.0	50.4	0.4	1.41
ь	2	64	25.0	24.9	-0.1	0.77
a+b		64	75.0	75.3	0.3	2.07
a-b		64	25.0	25.6	0.6	0.95
c		64	25.0	24.9	-0.1	0.78
d	:	64	5.0	4.5	-0.5	0.35
c+d		64	30.0	29.3	-0.7	0.92
c-d		64	20.0	20.4	0.4	0.78

s.d.(AB): Sw(within run): 0.67 S(between runs): 1.14 S/Sw: 1.69 s.d.(CD): Sw(within run): 0.55 S(between runs): 0.60 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

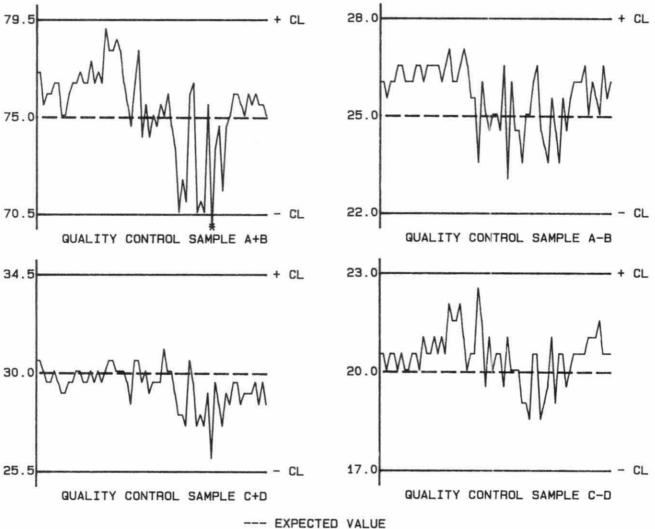
70.5 to 79.5 for A+B 22.0 to 28.0 for A-B 25.5 to 34.5 for C+D 17.0 to 23.0 for C-D

DUPL ICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Spa	n	s.d.	of var.(%)
	21	0.0 -	5.0	0.37	17.7
	35	5.0 -	10.0	0.69	9.2
	54	10.0 -	25.0	0.88	5.0
	49	25.0 -	50.0	0.97	2.5
	25	50.0 - 1	00.0	1.34	2.1
	184	Overal1		0.91	N/A

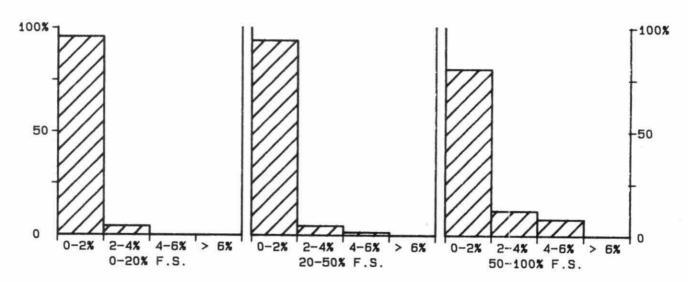
DETECTION CRITERION: 1.1

QUALITY CONTROL GRAPHS COLOUR-TRUE (TCU)

FROM: 03/01/85 TO: 31/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 TCU

*** COLOUR - APPARENT ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced: 15/10/80

Supervisor

: F. Tomassini

: Hazen Units

Sample Type/Matrix: Streams, Lakes

SAMPLING:

Quantity Required: 75 mL

Container

: Polystyrene

ANALYTICAL PROCEDURE:

Apparent colour is measured colourimetrically in a system calibrated with acidified chloroplatinate standards. Colour is measured using a broadband blue filter. Turbidity effects are partially suppressed by using a broadband red filter. Apparent colour is calculated from the two absorbance measurements using an empirically derived equation. If colour reads >70 or turbidity reads >20, true colour is reported after settling plus dilution if required. Approximate absorbance: 0.05 at the 70 true colour level.

Units

INSTRUMENTATION:

Two colourimeters, one with broadband blue filter(400-450nm) and the other with broadband red filter(660-740nm). Colourimetric measurement is through a 4.0 cm light path.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 1

Detection Criterion (T):4

CALIBRATION:

BL plus 1 standard

CONTROLS:

Calibration : LTBL plus 2 standards,eg,QCA

NOTES:

Slope factor is changed whenever light source in a colourimeter is replaced. This is accomplished by analyzing 7 standards.

COLOR-APPARENT QUALITY CONTROL DATA FROM 09/01/85 TO 18/12/85

Lab: Dorset

Analytical Range: 4 to 100 HU

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	161	50	50	Ø	1.1
ь	:	161	10	10	Ø	0.6
a+b	1	161	60	59	-1	1.5
a-b	:	161	40	40	Ø	0.9

s.d.(AB): Sw(within run): 0.6 S(between runs):

0.9 S/Sw: 1.39

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 53 to 67 for A+B 35 to 45 for A-B

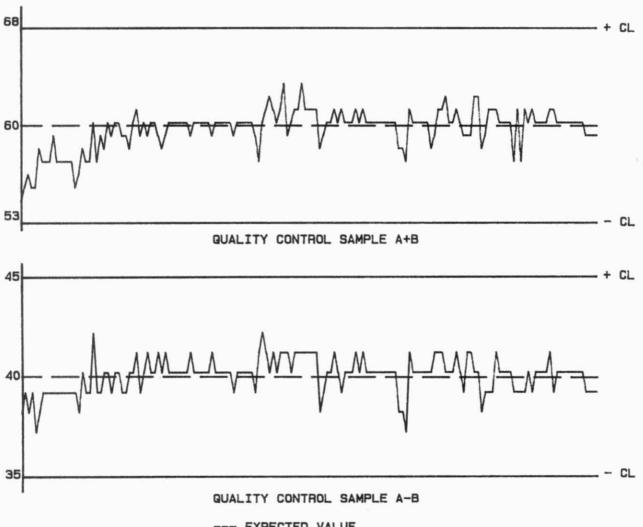
DUPL I CATES :	Number of	S	a.mp 1	e	Mean(2)	Coefficient
	Data Pairs	Con	cn S	pan	s.d.	of var.(%)
	54	0	-	10	1.2	20.1
	70	10	-	25	1.4	8.0
	151	25	-	50	1.6	4.5
	148	50	-	100	2.0	2.7
	423	0	vera	11	1.7	N/A

DETECTION CRITERION: 4

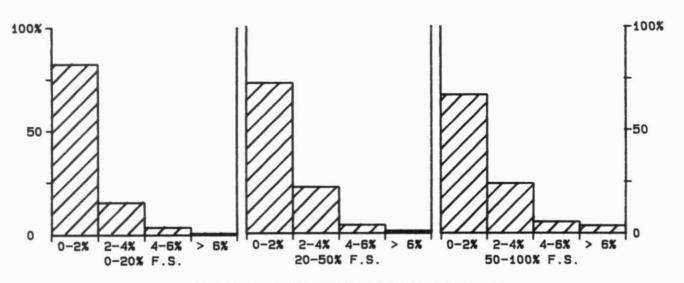
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	159	0	0.0

QUALITY CONTROL GRAPHS COLOR-APPARENT (HU)

FROM: 09/01/85 TO: 18/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 HU

*** COLOUR-TRUE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 28/02/84

LIS Test Name Code: COLTR Units : TCU
Work Station Code: ROCOL Unit Code : 342000

Method Code : 102BC9 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.05 at the 70 TCU level.

INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter(400-450nm). Turbitiy measurement is through a 5.0 cm. light path using a different broadband filter(660-740nm).

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 1.6

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg,QCA

Drift : BL plus 1 standard

NOTES:

New procedure was initiated to conform with change in "Ontario Drinking Water Objectives"; copy of research study is available on request.

22/08/85 -ROCOL workstation was phased out. Samples received subsequently were run on WCOL.

No data summary is available for period not covered in performance report.

COLOUR-TRUE QUALITY CONTROL DATA FROM 05/02/85 TO 22/08/85

Lab: Rivers and Lakes

Analytical Range: 1.6 to 100.0 TCU

CALL	DDAT	LACT	CONTROL
CHLI	DKUI	LUIA	CONTROL.

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	27	50.0	50.9	0.9	1.40
b	:	27	25.0	25.0	0.0	0.40
a+b	E	27	75.0	76.0	1.0	1.47
a-b	:	27	25.0	25.9	0.9	1.45

s.d.(AB): Sw(within run): 1.03 S(between runs): 1.03 S/Sw: 1.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

70.5 to 79.5 for A+B 22.0 to 28.0 for A-B

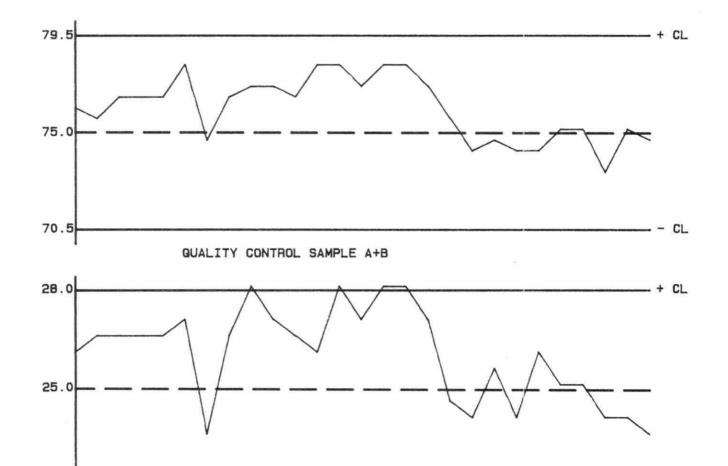
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	19	0.0 - 10.0	0.53	9.9
	27	10.0 - 25.0	0.51	2.9
	14	25.0 - 50.0	0.45	1.2
	7	50.0 - 100.0	0.33	0.4
	67	Overall	0.49	N/A

DETECTION CRITERION: 1.6

OTHER CHECKS:		Number	Data	Standard(1)
5		of Data	Me a.n	Deviation
STD.CAL	1	27	414	49.4
Long Term Blank	1	56	0.0	0.07

QUALITY CONTROL GRAPHS COLOUR-TRUE (TCU)

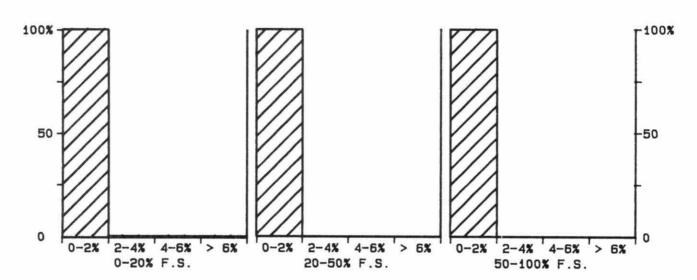
FROM: 05/02/85 TO: 22/08/85



QUALITY CONTROL SAMPLE A-B

22.0

--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 100 TCU

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '74

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : WPC Unit Code : 350351 : 002AI2 Method Code

Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Leachates

SAMPLING:

Quantity Required: 75 mL

: Glass or plastic Container

ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell and equilibrated to 25 C. The conductivity is read directly from a digital display.

INSTRUMENTATION

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

REPORTING:

Maximum Significant Figures: 4

Minimum Increment (W): 1

Detection Criterion (T): 3

CALIBRATION:

Standard resistor

CONTROLS:

Calibration: BL plus 3 standards, eg, QCA

CONDUCTIVITY QUALITY CONTROL DATA FROM 02/01/85 TO 30/12/85

Lab: Domestic Water

Analytical Range: 3 to 5000 uS/cm

COL	TODAT	TONE	CONTROL
unu	IDKUI	TUR	CUNTRUL

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	183	1399	1401	2	10.2
b		183	477	479	2	4.6
a+b	:	183	1876	1880	4	13.7
ab	1	183	922	923	i	8.0
c	:	183	477	479	2	4.6
d	:	183	193	195	2	1.7
c+d	:	183	670	674	4	5.7
c-d	E	183	284	284	Ø	3.8

s.d.(AB): Sw(within run): 5.7 S(between runs): 7.9 S/Sw: 1.40 s.d.(CD): Sw(within run): 2.7 S(between runs): 3.5 S/Sw: 1.29

On any given day the calibration is accepted if the values obtained lie within the ranges:

1801 to 1951 for A+B 872 to 972 for A-B 640 to 700 for C+D 264 to 304 for C-D

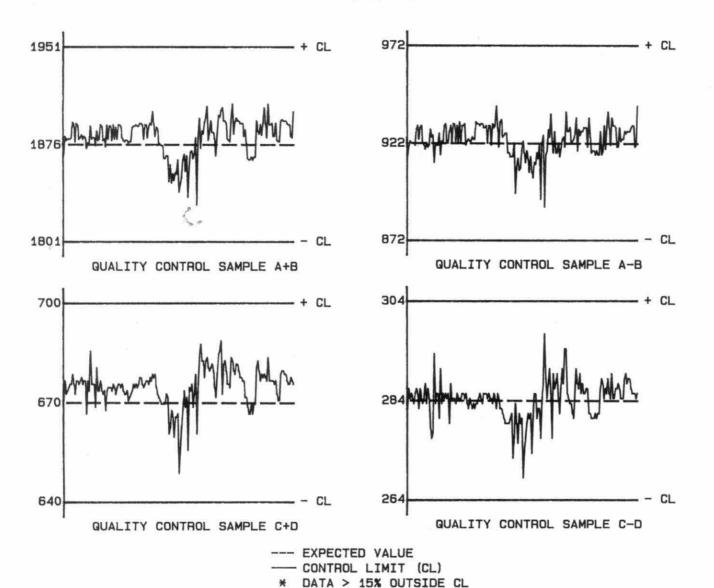
DUPLICATES:	Number of	S	amp	le	Mean(2)	Coefficient
	Data Pairs	Con	cn	Span	s.d.	of var.(%)
	36	Ø	-	130	1.0	1.3
	213	130	-	500	1.5	0.5
	129	500	-	1300	2.2	0.3
	16	1300	-	2500	2.5	0.1
	10	2500	-	5000	19.1	0.6
	404	0	ver	a11	3.5	N/A

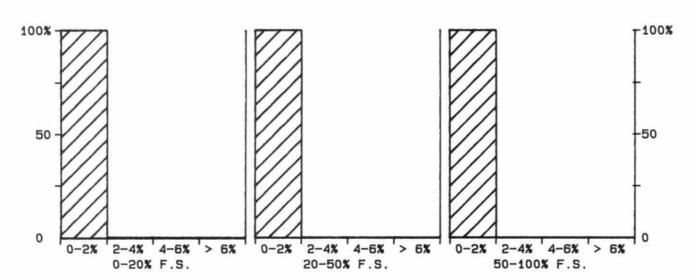
DETECTION CRITERION: 3

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Std Resistor	:	183	1590	0.0

QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 02/01/85 TO: 30/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5000 US/CM

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory : Dorset Methods Introduced: 01/06/76

Supervisor : F. Tomassini Units : uS/cm at 25 C

Sample Type/Matrix: Streams, Lakes, Precipation

SAMPLING:

Quantity Required: 75 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell and equilibrated to 25 C. The conductivity is read directly from a digital display.

INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

REPORTING:

Maximum Significant Figures: 4

Minimum Increment (W): 0.1 Detection Criterion (T): 1.3*

CALIBRATION:

None

CONTROLS:

Calibration: BL plus 2 standards, eg,QCA

NOTES:

*T value is based on duplicate analyses at concentrations above the lowest range.

CONDUCTIVITY QUALITY CONTROL DATA FROM 09/01/85 TO 16/12/85

Lab: Dorset

Analytical Range: N/A to 300 uS/cm

CALIBRATION CONTROL	CALI	BRAT	HOI	CON	TROL
---------------------	------	------	-----	-----	------

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	164	290	292	2	1.4
ь	1	164	74	75	1	0.5
a+b	:	164	364	367	3	1.5
a-b		164	216	217	1	1.4

s.d.(AB): Sw(within run): 1.0 S(between runs): 1.1 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 351 to 377 for A+B 207 to 225 for A-B

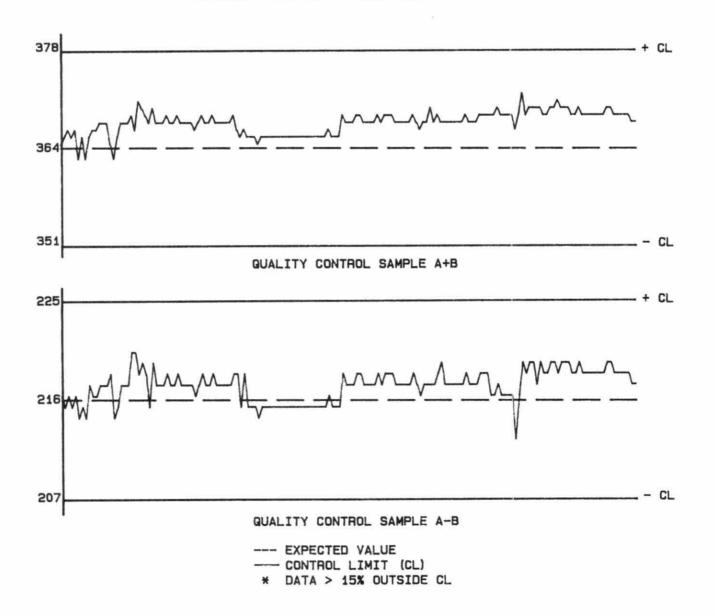
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	Ø	0.0 - 1	0.0 N/A	N/A
	9	10.0 - 2	0.0 0.43	2.4
	361	20.0 - 5	0.0 0.47	1.4
	27	50 - 10	0 0.6	0.9
	38	100 - 30	0 1.1	0.8
	435	Overall	0.6	N/A

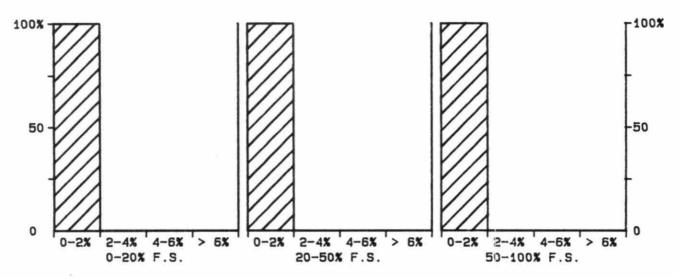
DETECTION CRITERION: N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Long Term Blank	:	163	1	0.3

QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 09/01/85 TO: 16/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 300 US/CM

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/04/78

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : PRICI Unit Code : 350351

Method Code : 002A12 Supervisor : M Rawlings

Sample Type/Matrix: Precipitation, Throughfall. Stemflow

SAMPL ING:

Quantity Required: 15 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

After equilibraton at 25 C, the conductivity of the sample is measured.

INSTRUMENTATION:

Automated modular continuous flow conductivity system comprised of sampler, water bath, conductivity meter with cell, chart recorder.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.1

Detection Criterion (T): 0.9

CALIBRATION:

Compatability between conductivity meter and chart recorder is confirmed by checking 3 standard resistances

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA

Drift : 1 solution every 10 samples

MODIFICATIONS:

18/10/83 -Automated continuous flow system was introduced.

NOTES:

A calibration standard for the ion chromatographic system is utilized as a drift control for the conductivity system, but its theoretical conductivity is unKnown.

CONDUCTIVITY QUALITY CONTROL DATA FROM 02/01/85 TO 19/12/85

Lab: Precipitation

Analytical Range: 0.9 to 100.0 uS/cm

	CONTROL

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	129	44.5	46.1	1.6	0.95
b		129	7.5	8.8	1.3	0.77
a+b	:	129	52.0	54.8	2.8	1.47
a-b	1	129	37.0	37.3	0.3	0.91

s.d.(AB): Sw(within run): 0.64 S(between runs): 0.86 S/Sw: 1.34

On any given day the calibration is accepted if the values obtained lie within the ranges:

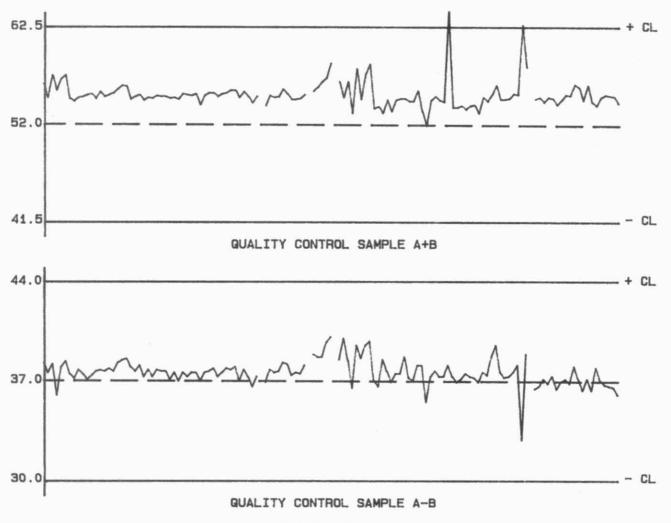
41.5 to 62.5 for A+B 30.0 to 44.0 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	13	0.0 - 10.0	0.31	4.0
	21	10.0 - 20.0	0.55	3.8
	77	20.0 - 50.0	1.16	3.4
	28	50.0 - 100.0	3.15	4.5
	139	Overall	1.67	N/A

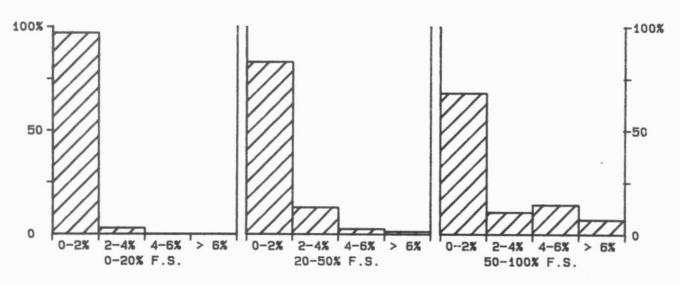
DETECTION CRITERION: 0.9

QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 02/01/85 TO: 19/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 100 US/CM

*** CONDUCTIVITY ***

IDENTIFICATION:

Method Introduced: 01/04/74 Laboratory Rivers and Lakes

: uS/cm at 25 C LIS Test Name Code: COND25 Work Station Code: ROCONTUR,RATS Units

: 350351 Unit Code

: J. Crowther Supervisor : 002B12 Method Code

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents

SAMPLING:

Quantity Required: 25 mL

: Glass or plastic Container

ANALYTICAL PROCEDURE:

After equilibraton at 25 C, the conductivity of the sample is measured.

INSTRUMENTATION:

Automated modular continuous flow conductivity system comprising sampler, water bath, conductivity meter with cell, microcomputer.

REPORTING:

Maximum Significant Figures: 3

Detection Criterion (T): 2 Minimum Increment (W):

CONTROLS:

Calibration: LTBL plus 4 standards, eg, QCA

: In run standards throughout the run, diluted tap water (20%V/V) Drift

MODIFICATIONS:

01/04/84 -Automated system introduced for conductivity range 20-1000 uS/cm. 09/05/85 -Analytical stream is not flowing during conductivity measurement. Radiometer conductivity meter was changed from model CDM 3 to model CDM 83. analytical range was expanded: 1 to 3000 uS/cm. This expansion was feasible due to the use of the auto-ranging CDM 83 module.

NOTES:

No data summary is available for period not covered in performance report.

CONDUCTIVITY QUALITY CONTROL DATA FROM 14/03/85 TO 31/12/85

Lab: Rivers and Lakes

Analytical Range: 2 to 2000 uS/cm

	FREAT	14017	COLE	mon .
LHL	IBKM	IIUN	LUN	TROL

		Number	Expected	Av.Concn	Av.Bias	Standard(1) Deviation
		of Data	Concn	Measured		Deviation
a		111	718	718	Ø	3.1
b	:	112	147	148	1	1.2
a+b	1	111	865	867	2	3.6
a-b	1	111	571	570	-1	3.0
c	:	112	147.0	148.5	1.5	1.24
d	1	112	37.1	38.3	1.2	0.39
c+d		112	184.1	186.8	2.7	1.51
c-d		112	109.9	110.2	0.3	1.05

s.d.(AB): Sw(within run): 2.1 S(between runs): 2.4 S/Sw: 1.11 s.d.(CD): Sw(within run): 0.74 S(between runs): 0.92 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

775 to 955 for A+B 511 to 631 for A-B 178.1 to 190.1 for C+D 105.9 to 113.9 for C-D

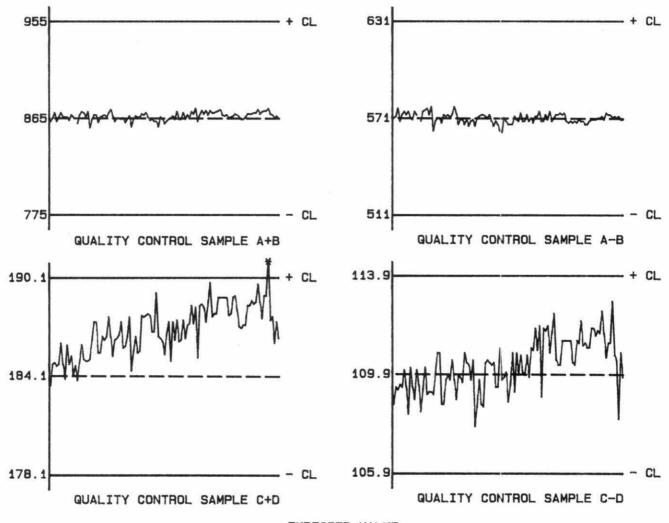
DUPLICATES:	Number of	S	amp	le	Mean(2)	Coefficient
	Data Pairs	Con	-n	Span	s.d.	of var.(%)
	57	0.0	-	50	0.7	2.0
	86	50	_	200	0.8	0.8
	102	200	_	500	1.4	0.4
	60	500	-	1000	1.9	0.3
	11	1000	-	2000	2.5	0.2
	316	0	ver	all	1.3	N/A

DETECTION CRITERION: 2.1

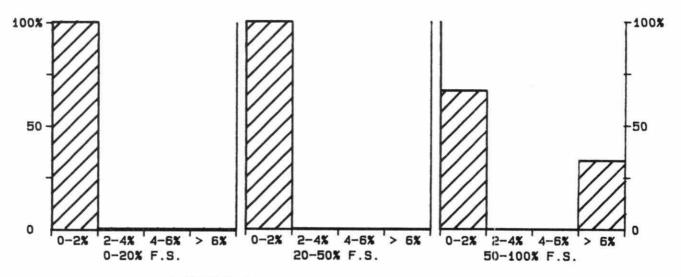
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Me an	Deviation
Cell Const	: 10	3.28	0.000

QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 14/03/85 TO: 31/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 3000 US/CM

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '74

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : COND-SEW Unit Code : 350351
Method Code : 002AI2 Supervisor : P. Campbell

Sample Type/Matrix: Sewage, Industrial Waste, Effluents

SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

The filtered sample is introduced into a jacketed conductivity cell and equilibrated to 25 C. The conductivity is read directly from an analog display.

equilibrated to ES C. The conductivity is read directly from an analog displa

INSTRUMENTATION

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 1 Detection Criterion (T): 4

CALIBRATION:

None

CONTROLS:

Calibration: BL plus 3 standards, eg, QCA

CONDUCTIVITY QUALITY CONTROL DATA FROM 04/01/85 TO 23/12/85

Lab: Sewage and Industrial Waste

Analytical Range: 4 to 3000 uS/cm

COL	TODAT	TONE	CONT	TOOL .
Lane	IBRAT	LUIN	CUIN	I IKUL *

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	38	1413	1410	-3	4.1
b	:	38	717.8	717.6	-0.2	2.15
a+b	:	38	2130.8	2127.3	-3.5	5.54
a-b	:	38	695.2	692.1	-3.1	3.56

s.d.(AB): Sw(within run): 2.5 S(between runs): 3.3 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 2040.8 to 2220.8 for A+B 635.2 to 755.2 for A-B

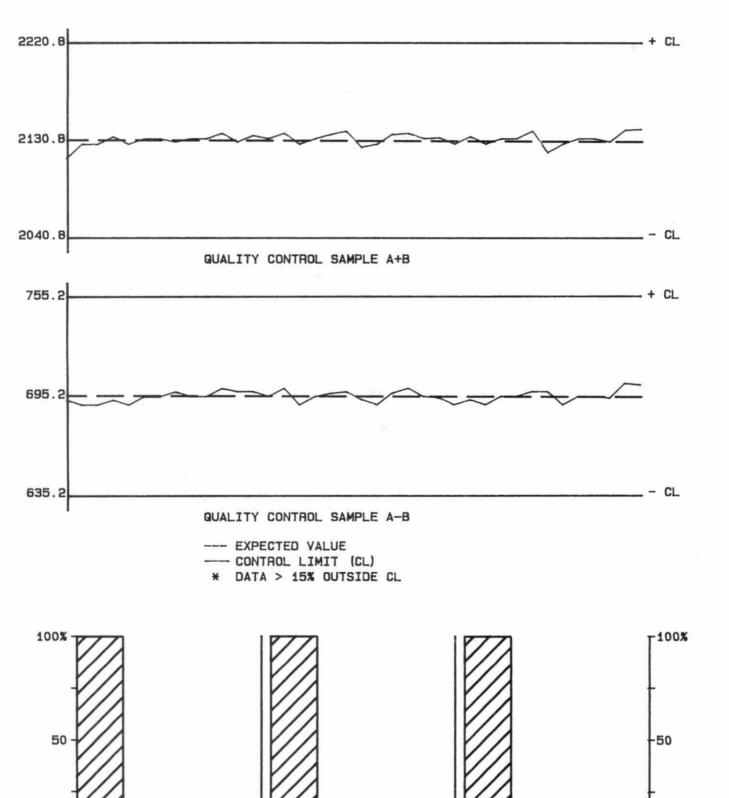
DUPLICATES:	Number of Data Pairs		amp cn :	le Span	Mean(2) s.d.	Coefficient of var.(%)
	22	Ø	-	500	1.2	0.4
	28	500	-	1000	0.8	0.1
	15	1000	-	1500	4.1	0.3
	1	1500	-	3000	N/A	N/A
	66	0	ver.	a11	2.1	N/A

DETECTION CRITERION: 4

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
	1			
Long Term Blank	4	38	1.20	0.572

QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 04/01/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 3000 US/CM

0-2% 2-4% 4-6% > 6%

20-50% F.S.

0-2% 2-4% 4-6% > 6%

50-100% F.S.

2-4% 4-6% > 6%

0-20% F.S.

*** FLUORIDE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/03/78
LIS Test Name Code: FFIDUR Units : ug/L as F
Work Station Code : WSPF Unit Code : 063809
Method Code : 001AIE Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Lakes, Streams

SAMPLING:

Quantity Required: 50 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

Fluoride is determined via an automated flow system for which the detector is a specific ion electrode; prior to measurement the sample is mixed with a high ionic strength buffer containing: sodium citrate, disodium ethylenediaminetetraacetate(EDTA), phosphoric acid, and sufficient sodium hydroxide to obtain pH 6.7

INSTRUMENTATION:

Automated modular continuous flow ion specific electrode system.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 1.4

CALIBRATION:

BL plus 1 standard in duplicate

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 1 standard

Interference: Combined fluoride and aluminum standard confirms that aluminum is not an interference.

MODIFICATIONS:

01/03/82- The above procedure is not described in HAMES, but a copy of the development report is available on request. The manual procedure in HAMES for the determination of fluoride by specific ion electrode is similar.

NOTES:

At the present time this procedure is restricted to special projects.

FLUORIDE QUALITY CONTROL DATA FROM 16/01/85 TO 24/12/85

Lab: Domestic Water

Analytical Range: 1.4 to 70.0 ug/L as F

COL	TAGGI	TONE	COLD	rpol .
LnL.	IBRAT	IUN	LUN	RUL

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	77	48.0	48.3	0.3	0.64
b	E	77	24.0	24.2	0.2	0.74
a+b	1	77	72.0	72.4	0.4	1.16
a-b	:	77	24.0	24.1	0.1	0.76

s.d.(AB): Sw(within run): 0.54 S(between runs): 0.69 S/Sw: 1.29

On any given day the calibration is accepted if the values obtained lie within the ranges:

68.3 to 75.7 for A+B 21.6 to 26.5 for A-B

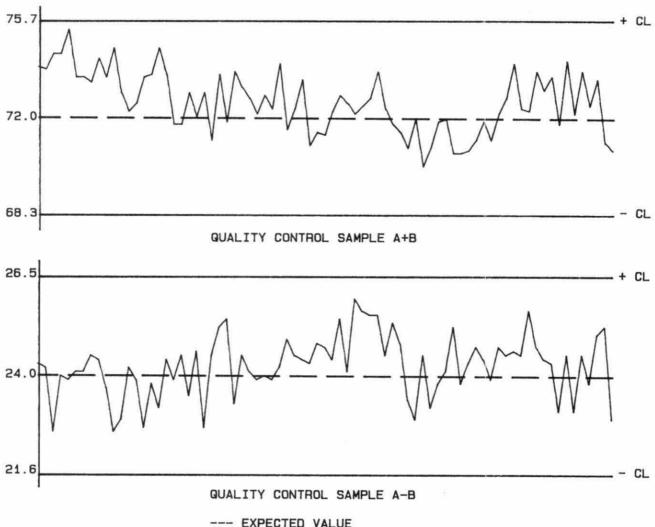
DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	3	0.0 - 1	0.0 0.48	7.1
	10	10.0 - 3	0.0 0.82	4.2
	119	30.0 - 5	0.0 0.81	1.9
	63	50.0 - 7	0.0 0.85	1.5
	195	Overall	0.82	N/A

DETECTION CRITERION: 1.4

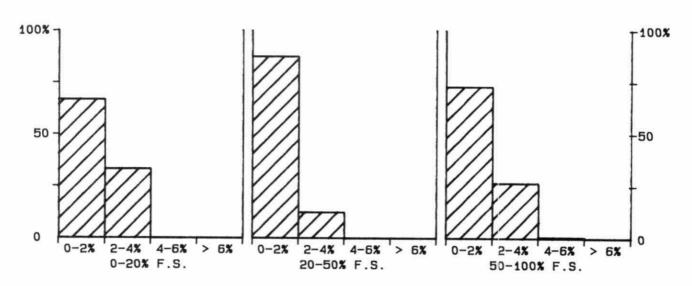
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
60ug/L F+500ug/L A1	:	76	60.1	1.00

QUALITY CONTROL GRAPHS FLUORIDE (UG/L AS F)

FROM: 16/01/85 TO: 24/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 70 UG/L AS F

90 *** FLUORIDE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '74 LIS Test Name Code: FFIDUR Units : mg/L as F

Work Station Code: WFNO3 or WFF Unit Code: 064809
Method Code: 003AC2 Supervisor: M. Rawl

Method Code : 003AC2 Supervisor : M. Rawlings Sample Type/Matrix: Domestic Waters, Surface Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160 C; the distillate is then reacted (in an acetic acid-acetate buffer media) with Alizarin Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.

Approximate absorbance is 0.6 at the 2.0 mg/L level

INSTRUMENTATION:

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm light path at 630 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.03

CALIBRATION:

BL plus 1 standard in duplicate

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 2 standards

FLUORIDE QUALITY CONTROL DATA FROM 02/01/85 TO 27/12/85

Lab: Domestic Water

Analytical Range: 0.03 to 2.00 mg/L as F

COL	TODAT	LACE	CONTRO	
I.PIL.	IPSPERI	111174	LININER	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	92	1.50	1.48	-0.02	0.028
b	1	92	0.30	0.31	0.01	0.013
a+b	1	92	1.80	1.78	-0.02	0.034
a-b	:	92	1.20	1.17	-0.03	0.027

s.d.(AB): Sw(within run): 0.019 S(between runs): 0.022 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

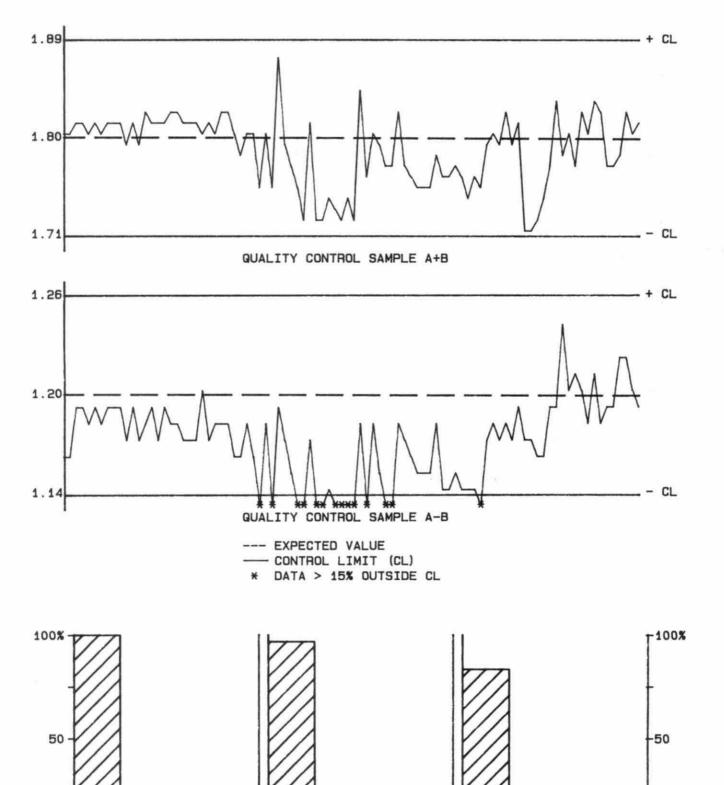
1.71 to 1.89 for A+B 1.14 to 1.26 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	174	0.00 - 0.20	0.009	10.6
	40	0.20 - 0.50	0.010	3.3
	24	0.50 - 1.00	0.011	1.3
	17	1.00 - 2.00	0.019	1.5
	255	Overall	0.010	N/A

DETECTION CRITERION: 0.03

QUALITY CONTROL GRAPHS FLUORIDE (MG/L AS F)

FROM: 02/01/85 TO: 27/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 MG/L AS F

20-50% F.S.

4-6% '> 6%

0-2%

2-4%

50-100% F.S.

4-6% > 6%

2-4%

0-2%

2-4% 4-6%

0-20% F.S.

> 6%

0-2%

*** IRON - TOTAL ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 20/04/76
LIS Test Name Code: FEUT Units : mg/L as Fe
Work Station Code: WFEMN Unit Code : 064826
Method Code : 504BC2 Supervisor : M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Sewage, Industrial Waste,
Effluents

LITTUELLES

SAMPLING:

Quantity Required: 100 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples(25.0 mL) are autoclaved in sulphuric acid-hydroxylamine media at 121 C for 45 min. The iron content of the digestate is determined colourimetrically by formation of the ferrous-2,4,6-Tri(2'pyridyl)-1,3,5-triazine(TPTZ) complex in a buffered system.

Approximate absorbance : 0.5 at the 2.0 mg/L level.

N.B. Manganese is determined simultaneously.

INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.02

CALIBRATION:

BL plus 1 undigested standard

CONTROLS:

Calibration: LTBL plus 2 undigested standards,eg,QCA
Recovery: Digested BL plus 2 digested standards,eg,R1

Drift : BL plus 1 undigested standard

NOTES:

Calibration standards are prepared from a hydrate: Fe(NH4)2(SO4)2.6H2O. Results are corrected using a digested blank value.

01/07/85 Test transferred to I.T.C. No data summary is available for period not covered in performance report.

IRON-TOTAL QUALITY CONTROL DATA FROM 02/01/85 TO 28/06/85

Lab: Domestic Water Analytical Range: 0.02 to 2.00 mg/L as Fe

CAL	TRRAT	MOL	CONTR	I.IO
~ 11	4017111	2011		

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured	s	Deviation
a	:	59	1.40	1.41	0.01	0.014
ь	E	59	0.28	0.30	0.02	0.013
a+b	:	59	1.68	1.71	0.03	0.023
a-b	:	59	1.12	1.11	-0.01	0.015

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.014 S/Sw: 1.27

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.59 to 1.77 for A+B 1.06 to 1.18 for A-B

0.011

N/A

RECOVER IES:		Number of Data		Conc		Av.Conc. Measured	Standard(1) Deviation
r1		59			1.40	1.40	0.046
r2	:	59			0.28	0.30	0.010
DUPLICATES:	Numbe Data	r of Pairs	0.000	mple n Sp		Mean(2) s.d.	Coefficient of var.(%)
	82	2	0.00	-	0.20	0.007	9.2
	38	3	0.20	-	0.50	0.013	3.9
	31		0.50	-	1.00	0.016	2.4
	16	1	1.00	-	2.00	0.017	1.1

DETECTION CRITERION: 0.02

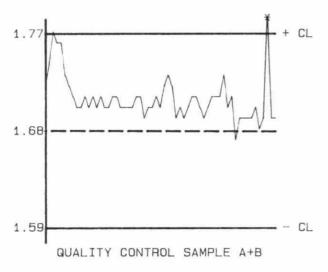
161

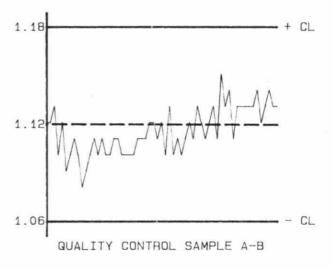
OTHER CHECKS:		Number	Data	Standard(1)
	0	f Data	Mean	Deviation
	-			
Digested Blank	:	59	0.01	0.006

Overall

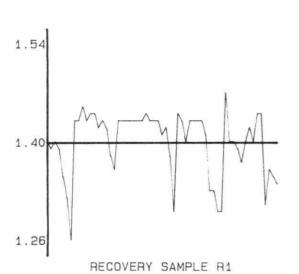
QUALITY CONTROL GRAPHS IRON-TOTAL (MG/L AS FE)

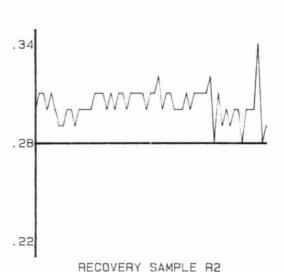
FROM: 02/01/85 TO: 28/06/85



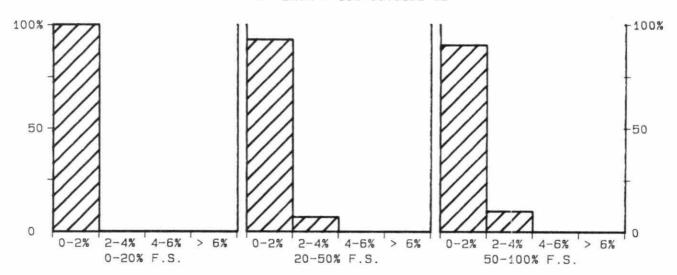


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS FE

*** MAGNESIUM ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/07/82
LIS Test Name Code: MGUR Units : mg/L as Mg

Work Station Code: WCAMGH Unit Code: 064812

Method Code : 001AA1 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 100 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.15 at the 20 mg/L level

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.3

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

MODIFICATIONS:

01/07/82- The method introduced on this date differed slightly from Method B for magnesium in HAMES in that full scale was 20.0 mg/L; concentrations of QC standards were also adjusted.

MAGNESIUM QUALITY CONTROL DATA FROM 02/01/85 TO 30/12/85

Lab: Domestic Water Analytical Range: 0.3 to 80.0 mg/L as Mg

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	133	52.0	52.1	0.1	0.73
b	:	133	13.0	13.4	0.4	0.35
a+b		133	65.0	65.5	0.5	0.83
a-b	1	133	39.0	38.8	-0.2	0.78
c		133	13.0	13.2	0.2	0.22
d	:	133	2.6	2.6	-0.0	0.09
c+d	:	133	15.6	15.7	0.1	0.27
c - d	2	133	10.4	10.6	0.2	0.20

s.d.(AB): Sw(within run): 0.55 S(between runs): 0.57 S/Sw: 1.04 s.d.(CD): Sw(within run): 0.14 S(between runs): 0.17 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

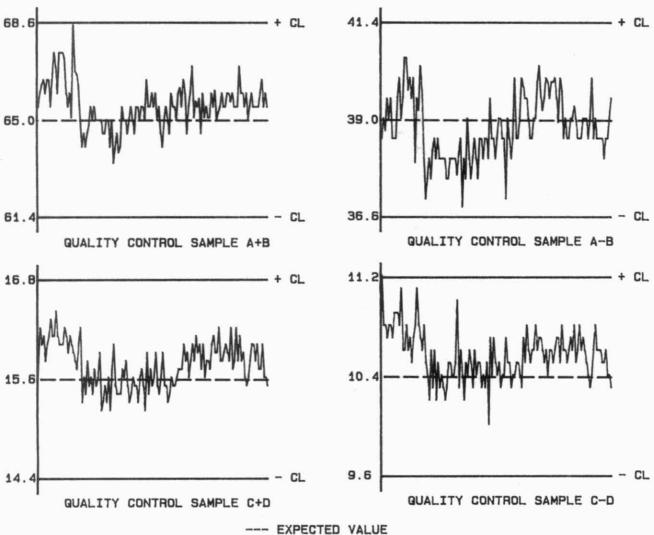
61.4 to 68.6 for A+B 36.6 to 41.4 for A-B 14.4 to 16.8 for C+D 9.6 to 11.2 for C-D

DUPL I CATES :	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Sp	an	s.d.	of var.(%)
	69	0.0 -	4.0	0.11	5.4
	105	4.0 -	10.0	0.13	1.8
	85	10.0 -	20.0	0.24	1.5
	91	20.0 -	40.0	0.38	1.4
	14	40.0 -	80.0	1.01	2.1
	364	Overal	1	0.31	N/A

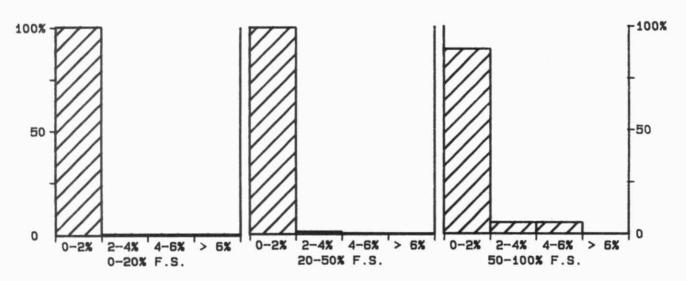
DETECTION CRITERION: 0.3

QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 02/01/85 TO: 30/12/85



--- EXPECTED VALUE
---- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): BO MG/L AS MG

*** MAGNESIUM ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 18/05/79

LIS Test Name Code: MGUR Units : mg/L as Mg Work Station Code: PRAA Unit Code : 064812

Work Station Code : PRAA Unit Code : 064812
Method Code : 001CA1 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 285.2 nm with an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the 0.50 mg/L level.

INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer(AAS) system

REPORTING:
Maximum Significant Figures: 3

Detection Criterion (T): 0.012

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Minimum Increment (W): 0.005

Drift : BL every 10 samples, 2 standards every 20 samples.

MODIFICATIONS:

17/05/85 - Three additional calibration standards were set up.

- Flow injection introduction of sample was adopted.

 System further automated with the addition of a microcomputer to co-ordinate sampler, injection, AAS "read", and data reduction.

- Sample required reduced to 5 mL.

MAGNESIUM QUALITY CONTROL DATA FROM 09/01/85 TO 20/12/85

Lab: Precipitation Analytical Range: 0.012 to 0.500 mg/L as Mg

indiffical Range. Dible to biobb ing/L as Pr

CALIBRATION C	ONTROL
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		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	68	0.300	0.299	-0.001	0.0079
ь	I	69	0.050	0.049	-0.001	0.0046
a+b	:	68	0.350	0.349	-0.001	0.0091
a-b	I	68	0.250	0.250	0.000	0.0092

s.d.(AB): Sw(within run): 0.0065 S(between runs): 0.0065 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

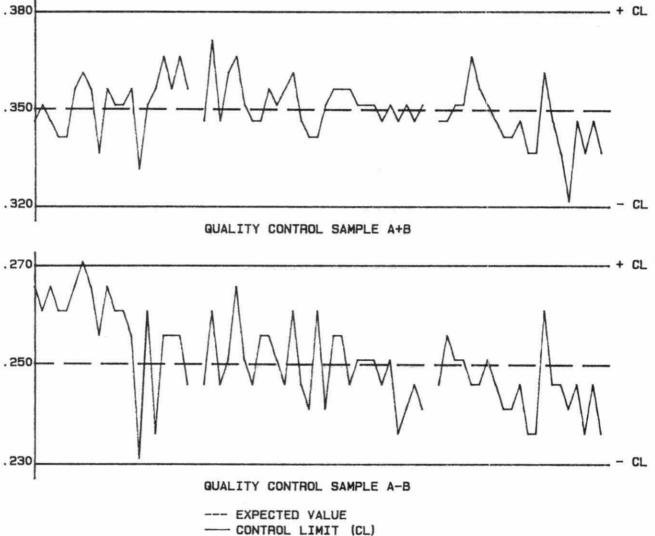
0.320 to 0.380 for A+B 0.230 to 0.270 for A-B

DUPL I CATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	91	0.000 - 0.050	0.0041	16.9
	35	0.050 - 0.100	0.0050	6.9
	25	0.100 - 0.250	0.0098	6.3
	15	0.250 - 0.500	0.0071	2.0
	166	Overall	0.0058	N/A

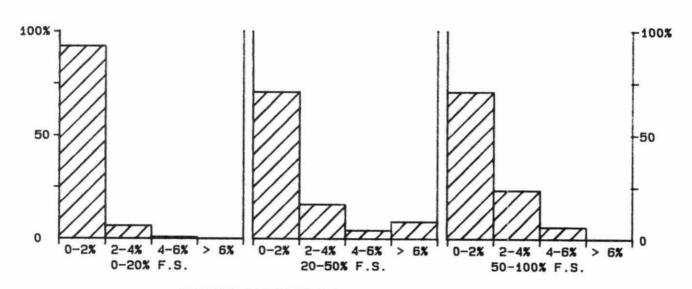
DETECTION CRITERION: 0.012

QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 09/01/85 TO: 20/12/85



DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .5 MG/L AS MG

*** MAGNESIUM ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: MGUR Units : mg/L as Mg

Work Station Code : RMAAS Unit Code : 064812

Method Code : 001AA1,001BA1 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: RMAAS: 1.25

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.01

Detection Criterion (T): 0.05,0.06,0.11

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA for each analytical range

Drift : BL plus 1 standard

MODIFICATIONS:

01/12/81- Calibration range became 5.00 mg/L full scale; second analytical range was dropped.

01/03/84- Analytical range(RMCAMGL) was added; full scale:1.00 mg/L. This range is currently restricted to special programs.

01/09/84- Analytical range(RMCAMGH) was increased from 5.00 to 10.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Calcium is no longer determined simultaneously.

25/09/85- Calibration range became 7.0 mg/L full scale; second analytical range was dropped. Microcomputer controlled system.

NOTES:

Three analytical ranges were used during 1985: 1.00, 7.00, and 10.0 mg/L as Mg full scale. Detection criteria above apply to these ranges respectively. *T value is based on duplicate analyses at concentrations above the lowest

range.

MAGNESIUM QUALITY CONTROL DATA FROM 03/01/85 TO 09/09/85

Lab: Rivers and Lakes

Analytical Range: 0.11 to 10.00 mg/L as Mg

CAL	IBRAT	KION	CONT	TPOL :
	LONI	1 1011	CULT	NOL

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
а.	:	37	7.50	7.55	0.05	0.136
b	:	37	2.50	2.52	0.02	0.078
a+b	:	37	10.00	10.07	0.07	0.177
a-b	:	37	5.00	5.02	0.02	0.133

s.d.(AB): Sw(within run): 0.094 S(between runs): 0.111 S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.55 to 10.45 for A+B 4.70 to 5.30 for A-B

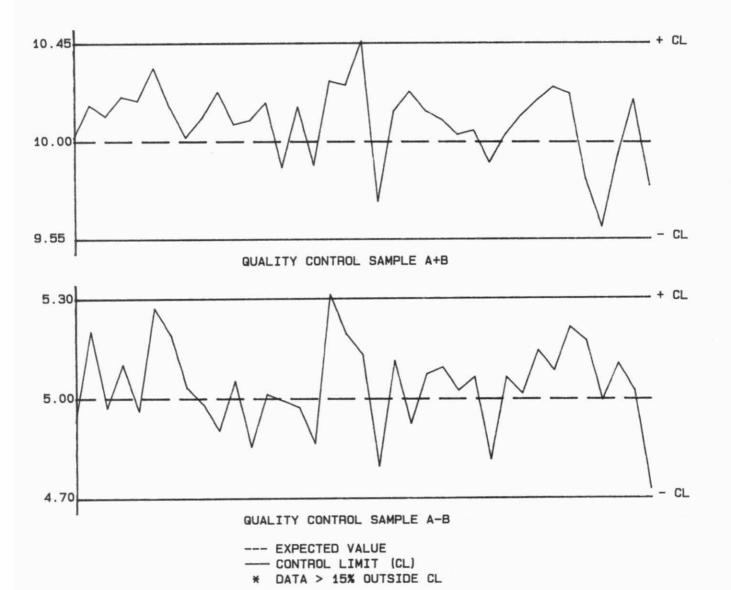
DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	18	0.00 - 0.50	0.038	10.2
	23	0.50 - 1.00	0.101	13.9
	16	1.00 - 2.50	0.077	4.8
	21	2.50 - 5.00	0.380	10.0
	10	5.00 - 10.00	0.226	3.1
	88	Overall	0.210	N/A

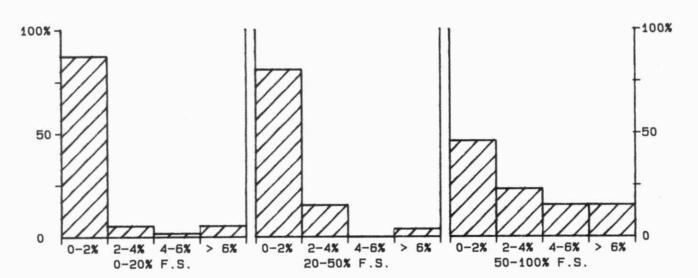
DETECTION CRITERION: 0.11

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	27	1.03	0.114
Long Term Blank	:	Ø	N/A	N/A

QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 03/01/85 TO: 09/09/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS MG

MAGNESIUM QUALITY CONTROL DATA FROM 02/10/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.06 to 7.00 mg/L as Mg

CALIBRA	TIO	N CONTROL: Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	:	25	5.60	5.61 0.49	0.01 0.00	0.082 0.013
b a+b	:	25 25	0.49 6.09	6.10	0.01	0.084
a-b	1	25	5.11	5.12	0.01	0.082
s.d.(AE	3):	Sw(within run): 0.058	S(between runs):	0.059	S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

5.77 to 6.40 for A+B 4.90 to 5.32 for A-B

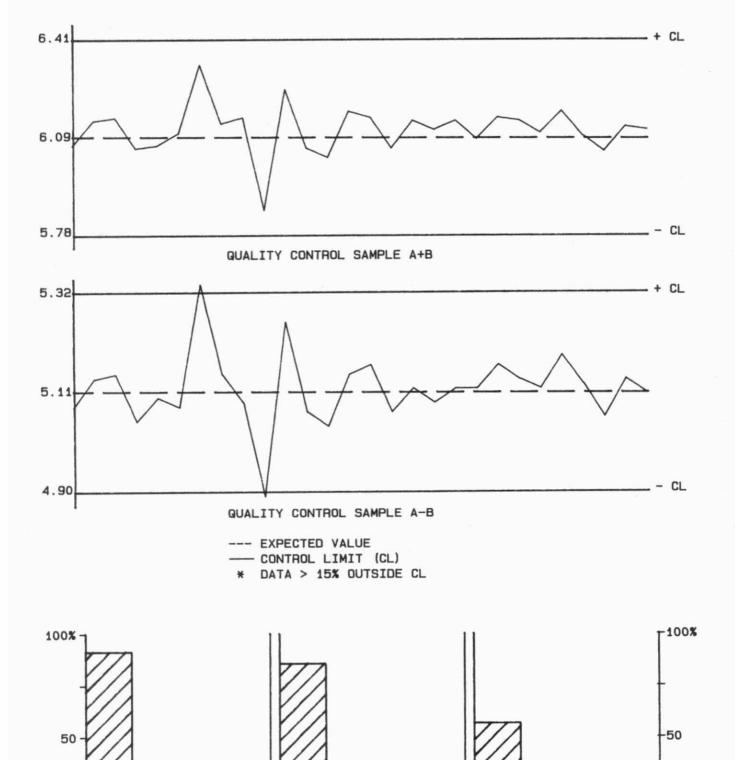
DUPLICATES:	Number of Data Pairs	Sample Concn Sp		Mean(2) s.d.	Coefficient of var.(%)
	3	0.00 -	0.35	0.021	6.1
	34	0.35 -	0.70	0.032	6.0
	20	0.70 -	1.40	0.032	3.6
	9	1.40 -	3.50	0.071	3.3
	7	3.50 -	7.00	0.402	8.0
	73	Overa	1 1	0.130	N/A

DETECTION CRITERION: 0.06

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	Ø	N/A	N/A
Long Term Blank	:	Ø	N/A	N/A

QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 25/09/85 TO: 30/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 7 MG/L AS MG

2-4% 4-6%

20-50% F.S.

> 6%

0-2%

50-100% F.S.

0-2%

0-2%

2-4%

4-6% 0-20% F.S.

MAGNESIUM QUALITY CONTROL DATA FROM 08/01/85 TO 10/09/85

Lab: Rivers and Lakes

Analytical Range: N/A to 1.00 mg/L as Mg

CALIBRATION	CONTROL

	Number	Expected	Av.Concn	Av.Bias	Standard(1)
	of Data	Concn	Measured		Deviation
:	41	0.75	0.75	0.00	0.013
:	41	0.25	0.25	-0.00	0.008
:	41	1.00	1.00	0.00	0.015
	41	0.50	0.50	0.00	0.015
	:	of Data : : 41 : 41	Number Expected of Data Concn : 41 0.75 : 41 0.25 : 41 1.00	Number Expected Av.Concn of Data Concn Measured : 41 0.75 0.75 : 41 0.25 0.25 : 41 1.00 1.00	Number Expected Av.Concn Av.Bias of Data Concn Measured : 41 0.75 0.75 0.00 : 41 0.25 0.25 -0.00 : 41 1.00 1.00 0.00

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.011 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.95 to 1.05 for A+B

0.47 to 0.53 for A-B

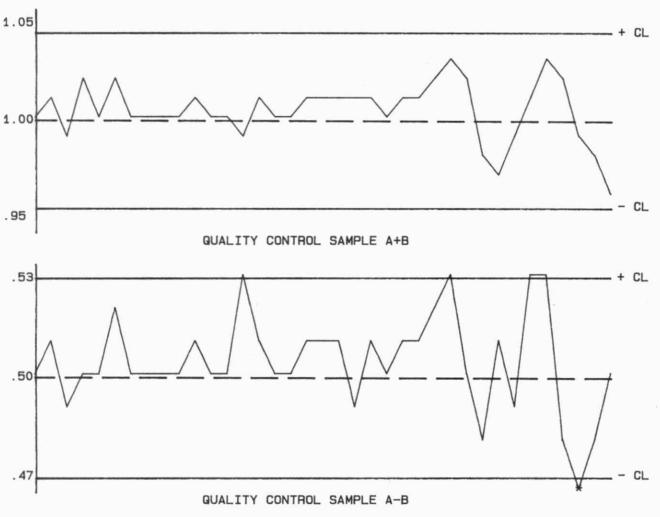
DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	1	0.00 - 0	.20 N/A	N/A
	17	0.20 - 0	.50 0.018	4.6
	76	0.50 - 1	.00 0.031	4.3
	94	Overall	0.029	N/A

DETECTION CRITERION: N/A

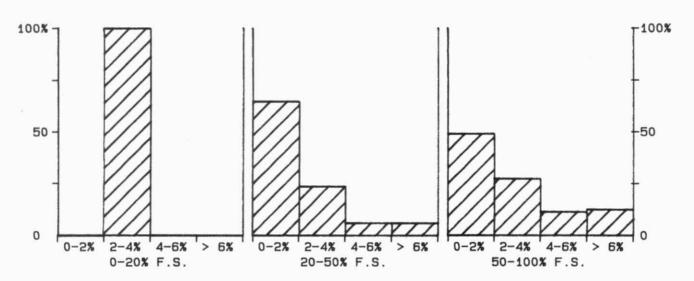
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance		37	1.03	0.199
Long Term Blank	:	Ø	N/A	N/A

QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 08/01/85 TO: 10/09/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1 MG/L AS MG

*** MANGANESE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/05/83

LIS Test Name Code: MNUT Units : mg/L as Mn Work Station Code: WFEMN Unit Code : 064825

Method Code : 504BC2 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Sewage, Leachates, Effluents

SAMPLING:

Quantity Required: 100 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples(25.0 mL) are autoclaved in sulphuric acid-hydroxylamine media at 121 C for 45 min. The manganese content of the digestate is determined colourimetrically by formation of the manganese-formaldoxime complex in a buffered system; this system is designed to suppress interferences from cations such as iron. A reference stream, based on an inverted order of reagent addition, is also required for suppression of cation interference.

Approximate absorbance: 0.3 at the 0.5 mg/L level.

N.B. Iron is determined simultaneously.

INSTRUMENTATION:

-Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a $5.0~\rm cm$. light path at $480~\rm nm$.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.002

Detection Criterion (T): 0.007

CALIBRATION:

BL plus 1 undigested standard

CONTROLS

Calibration: LTBL plus 2 undigested standards,eg,QCA Recovery: Digested BL plus 2 digested standards,eg,R1

Drift : BL plus 1 undigested standard

Interference: Iron standard confirms suppression of cation interference.

MODIFICATIONS:

01/05/83- The method introduced on this date differed from Method A for manganese in HAMES in that full scale was 0.5~mg/L. Concentrations of QC standards were also adjusted.

NOTES:

Calibration standards are prepared from a hydrate: MnCl2.4H2O Results are corrected using a digested blank value. 01/07/85 Test transferred to I.T.C. No data summary is available for period not covered in performance report.

MANGANESE QUALITY CONTROL DATA FROM 02/01/85 TO 28/06/85

Lab: Domestic Water Analytical Range: 0.007 to 0.500 mg/L as Mn

CAL I	BRAT	ION	CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	57	0.350	0.350	0.000	0.0020
ь	:	57	0.070	0.070	0.000	0.0016
a+b	:	57	0.420	0.421	0.001	0.0026
a-b		57	0.280	0.280	-0.000	0.0025

s.d.(AB): Sw(within run): 0.0018 S(between runs): 0.0018 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.390 to 0.450 for A+B 0.260 to 0.300 for A-B

RECOVERIES	Number of Data		a C	xpected oncn	Av.Conc. Measured	Standard(1) Deviation
r1 r2	:	57 57		0.350 0.070	0.352 0.072	0.0060 0.0019
DUPL ICATES:	Number of Data Pairs		Sam Concn	•	Mean(2) s.d.	Coefficient of var.(%)
	11:	В	0.000 - 0.050 - 0.100 - 0.250 -	0.100 0.250	0.0023 0.0031 0.0076 0.0040	13.3 4.5 4.6 1.2

Overall 0.0034

N/A

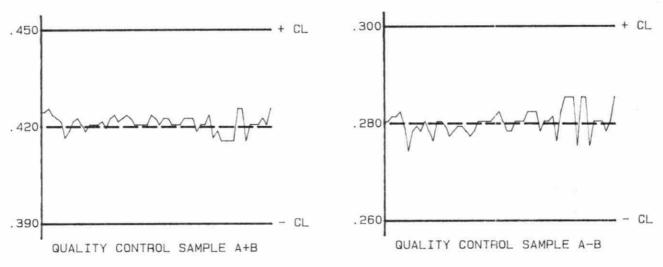
DETECTION CRITERION: 0.007

152

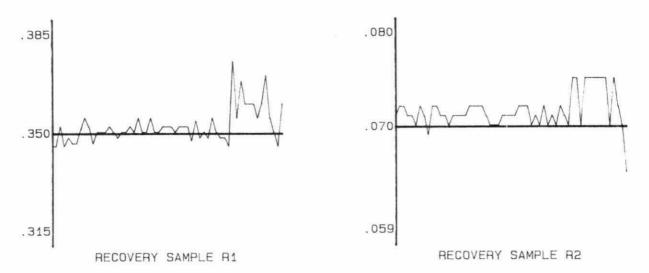
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Digested Blank	:	57	0.003	0.0016

QUALITY CONTROL GRAPHS MANGANESE (MG/L AS MN)

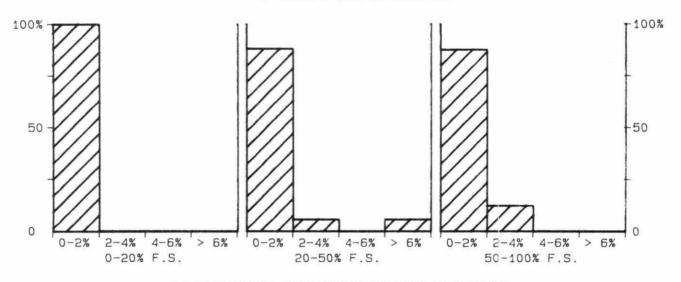
FROM: 02/01/85 TO: 28/06/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .5 MG/L AS MN

*** NITROGEN-AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/06/76
Supervisor : F. Tomassini Units : ug/L as N

Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL

Container : Plastic (polystyrene)

SAMPLE PREPARATION:

Samples are filtered through 0.45u membrane filters.

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the filtrate via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.40 at the 1000ug/L as N level. N.B. Nitrate plus nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 1

Detection Criterion (T): 3.5

CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL plus 1 standard

NITROGEN - AMMONIA + AMMONIUM QUALITY CONTROL DATA FROM 10/01/85 TO 19/12/85

Lab: Dorset

Analytical Range: 3.5 to 1000 ug/L as N

CALIBRA	TIO	N CONTROL:				
		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	48	750	752	2	6.0
b		48	250	249	- 1	5.3
a+b		48	1000	1001	1	9.1
a-b		48	500	503	3	6.7
, c	:	48	75.0	75.2	0.2	1.02
ď		48	25.0	25.0	0.0	0.97
		48	100.0	100.1	0.1	1.60
c-d	:	48	50.0	50.2	0.2	1.19
s.d.(AE	3):	Sw(within run): 4.7	S(between runs):	5.7	S/Sw: 1.19
s.d.(C	3):	Sw(within rur	0.84	S(between runs):	1.00	S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

970 to 1030 for A+B 480 to 520 for A-B 88.0 to 112.0 for C+D 42.0 to 58.0 for C-D

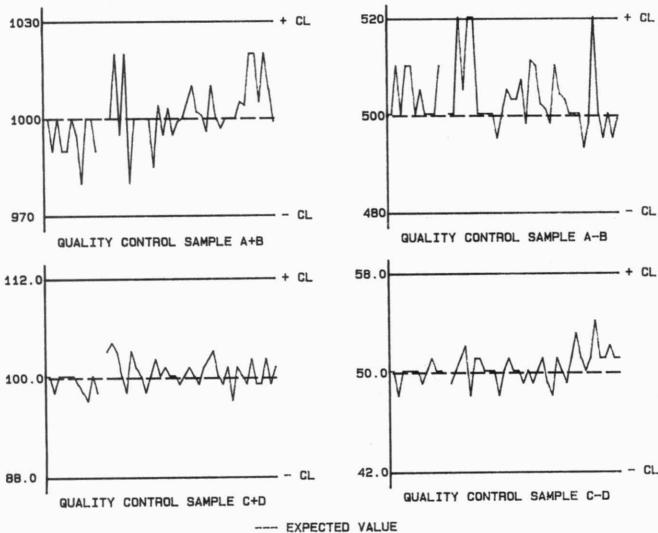
DUPLICATES:	Number of	NOONGESTAN NATA		Mean(2)	Coefficient
	Data Pairs			s.d.	of var.(%)
	78	0.0 -	25.0	1.17	14.0
	15	25.0 -	50.0	0.89	2.6
	10	50.0 -	100.0	2.41	3.6
	8	100 -	500	10.2	6.0
	1	500 -	1000	N/A	N/A
	112	Over	all	3.1	N/A

DETECTION CRITERION: 3.5

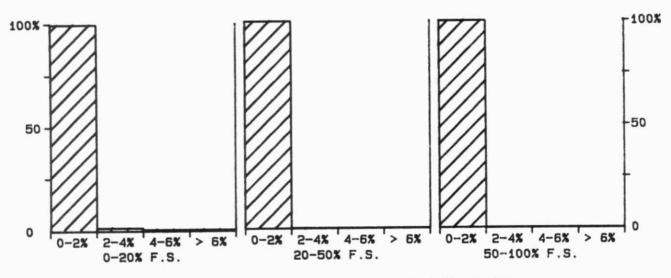
OTHER CHECKS:	Number of Data		Data Mean	Standard(1) Deviation
STD. CAL.	:	48	292	67.2
Long Term Blank	:	48	0.6	0.79

QUALITY CONTROL GRAPHS NITROGEN - AMMONIA + AMMONIUM (UG/L AS N)

FROM: 10/01/85 TO: 19/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UG/L AS N

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/05/84
LIS Test Name Code: NNHTFR,NNHTUR Units : mg/L as N
Work Station Code : PRNUT Unit Code : 064807
Method Code : 103CC2 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 1.1 at 5.00 mg/L as N level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 630 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.005

Detection Criterion (T): 0.014

CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards every 10 samples

MODIFICATIONS:

01/05/84- The procedure introduced on this date is the same as Method A for nitrogen-ammonia in HAMES except that the samples are not filtered and the full scale concentration is 5.00 mg/L as N.

NITROGEN - AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 04/01/85 TO 18/12/85

Lab: Precipitation Analytical Range: 0.012 to 5.00 mg/L as N

|--|

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	95	4.00	4.00	-0.00	0.026
b		95	0.80	0.81	0.01	0.013
a+b	1	95	4.80	4.80	0.00	0.032
a-b	:	95	3.20	3.19	-0.01	0.025
c	:	95	0.800	0.803	0.003	0.0054
d	1	95	0.200	0.192	-0.008	0.0045
c+d	:	95	1.000	0.995	-0.005	0.0074
c-d	2	95	0.600	0.612	0.012	0.0066

s.d.(AB): Sw(within run): 0.018 S(between runs): 0.021 S/Sw: 1.16 s.d.(CD): Sw(within run): 0.0047 S(between runs): 0.0050 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

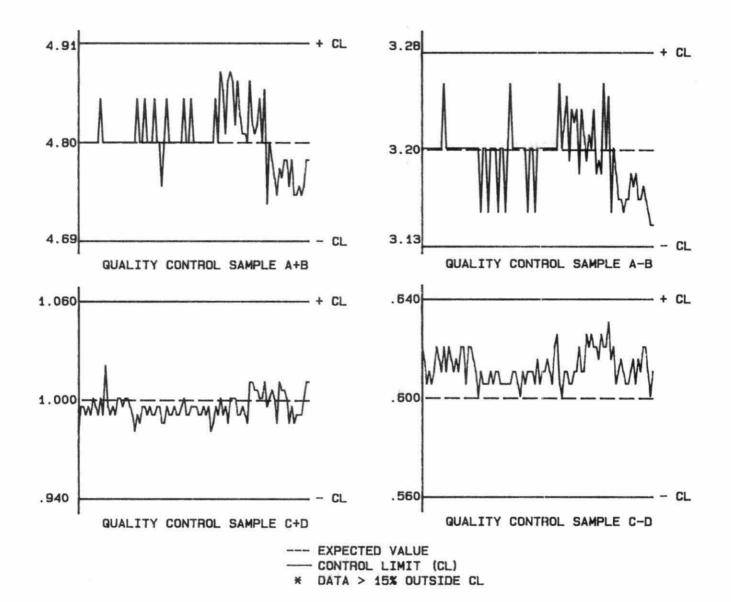
4.69 to 4.91 for A+B 3.13 to 3.28 for A-B 0.940 to 1.060 for C+D 0.560 to 0.640 for C-D

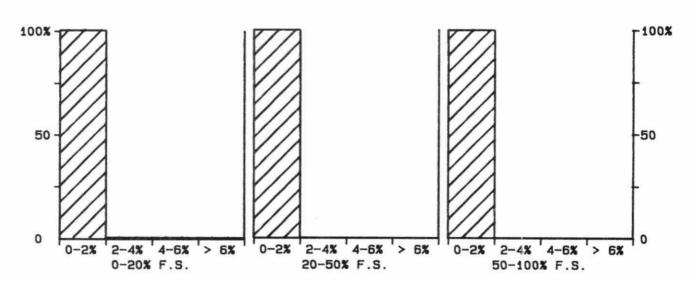
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	70	0.000 - 0.100	0.0039	11.7
	146	0.100 - 0.500	0.0031	1.2
	33	0.500 - 1.000	0.0077	1.1
	23	1.00 - 5.00	0.020	0.9
	272	Overall	0.007	N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Std. cal		95	739	123.4
Long Term Blank	:	94	0.003	0.0024

QUALITY CONTROL GRAPHS NITROGEN - AMMONIA PLUS AMMONIUM (MG/L AS N)

FROM: 04/01/85 TO: 18/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 5 MG/L AS N

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNHTFR Units : mg/L as N
Work Station Code: RNDNP Unit Code : 064807
Method Code: 103DC2 Supervisor: J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.5 at 2.00 mg/L as N level.

N.B. Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.002

Detection Criterion (T): 0.011

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus one standard

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

NITROGEN-AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

Lab: Rivers and Lakes

Analytical Range: 0.011 to 2.00 mg/L as N

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	137	1.60	1.62	0.02	0.013
b	1	137	0.40	0.41	0.01	0.007
a+b	:	137	2.00	2.03	0.03	0.014
a-b	:	137	1.20	1.21	0.01	0.016
c	:	137	0.400	0.408	0.008	0.0068
d	I	137	0.200	0.206	0.006	0.0046
c+d	I	137	0.600	0.613	0.013	0.0095
c-d	2	137	0.200	0.202	0.002	0.0067

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.010 S/Sw: 0.92 s.d.(CD): Sw(within run): 0.0047 S(between runs): 0.0058 S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

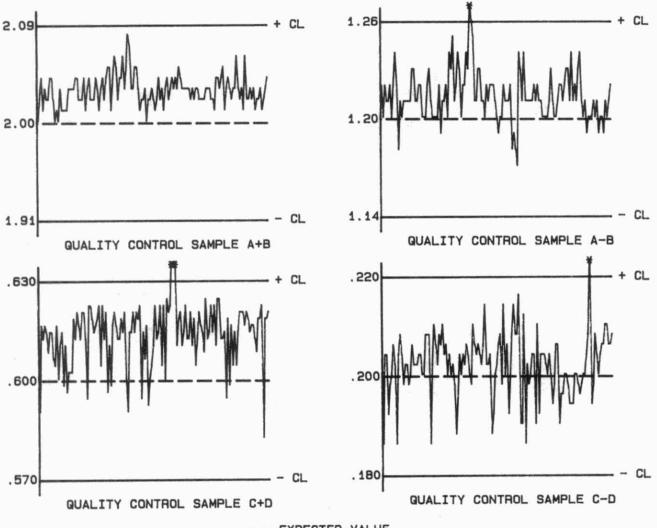
1.91 to 2.09 for A+B 1.14 to 1.26 for A-B 0.570 to 0.630 for C+D 0.180 to 0.220 for C-D

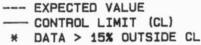
DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	188	0.000 - 0.040	0.0037	16.7
	120	0.040 - 0.100	0.0103	16.6
	39	0.100 - 0.200	0.0050	3.5
	16	0.200 - 0.400	0.0099	3.4
	18	0.40 - 2.00	0.026	2.9
	381	Overall	0.009	N/A

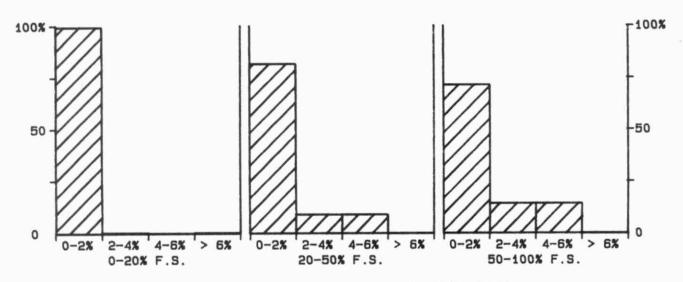
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me a.n	Deviation
Long Term Blank	:	136	0.009	0.0058

QUALITY CONTROL GRAPHS NITROGEN-AMMONIA PLUS AMMONIUM (MG/L AS N)

FROM: 03/01/85 TO: 20/12/85







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/77
LIS Test Name Code: NNHTFR Units : mg/L as N

Work Station Code : SNH3P Unit Code : 064807

Method Code : 103AC2 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance: 0.3 at 20.0 mg/L as N level. N.B. Reactive orthophosphate is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus one 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.15

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: LTBL plus 4 standards, eg, QCA

Drift : BL plus 2 standards

NITROGEN - AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 03/01/85 TO 30/12/85

Lab: Sewage and Industrial Waste Analytical Range: 0.15 to 50.0 mg/L as N

			 -
CAL	TDDAT	FIGNI	TROL:
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		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	142	35.0	35.6	0.6	0.47
b	:	142	14.0	14.4	0.4	0.21
a+b		142	49.0	50.0	1.0	0.62
a-b	:	142	21.0	21.2	0.2	0.39
c	:	143	14.00	14.13	0.13	0.240
d	:	143	3.50	3.69	0.19	0.159
c+d	1	143	17.50	17.81	0.31	0.354
c-d	1	143	10.50	10.44	-0.06	0.201

s.d.(AB): Sw(within run): 0.28 S(between runs): 0.36 S/Sw: 1.32 s.d.(CD): Sw(within run): 0.142 S(between runs): 0.204 S/Sw: 1.43

On any given day the calibration is accepted if the values obtained lie within the ranges:

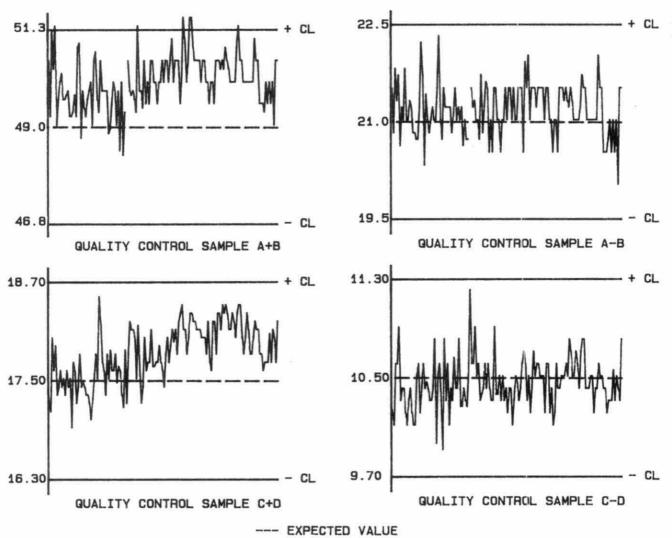
46.8 to 51.2 for A+B 19.5 to 22.5 for A-B 16.30 to 18.70 for C+D 9.70 to 11.30 for C-D

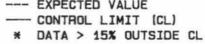
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	134	0.00 - 2.00	0.052	24.4
	14	2.00 - 5.00	0.154	4.1
	18	5.00 - 10.00	0.122	1.5
	36	10.0 - 20.0	0.15	1.0
	30	20.0 - 50.0	0.38	1.2
	232	Overal1	0.16	N/A

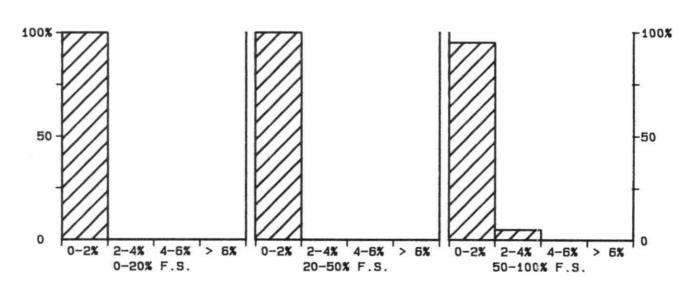
OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Me a.n	Deviation
Std. Cal	1	104	552	106.8
Long Term Blank	1	126	0.05	0.006

QUALITY CONTROL GRAPHS NITROGEN - AMMONIA PLUS AMMONIUM (MG/L AS N)

FROM: 03/01/85 TO: 30/12/85







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS N

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/04/78
LIS Test Name Code: NNO3UR Units : mg/L as N

Work Station Code : PRICI Unit Code : 064807

Method Code : 003AIO Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

SAMPLING:

Quantity Required: 15 mL

Container : Polyethylene

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 10 uS/cm

N.B. Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.05

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : 1 standard every 10 samples

NITROGEN-NITRATE QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Precipitation Analytical Range: 0.05 to 2.00 mg/L as N

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	140	1.60	1.60	0.00	0.013
b	:	139	0.40	0.39	-0.01	0.014
a+b	:	139	2.00	2.00	-0.00	0.019
ab	:	139	1.20	1.21	0.01	0.019

s.d.(AB): Sw(within run): 0.013 S(between runs): 0.014 S/Sw: 1.01

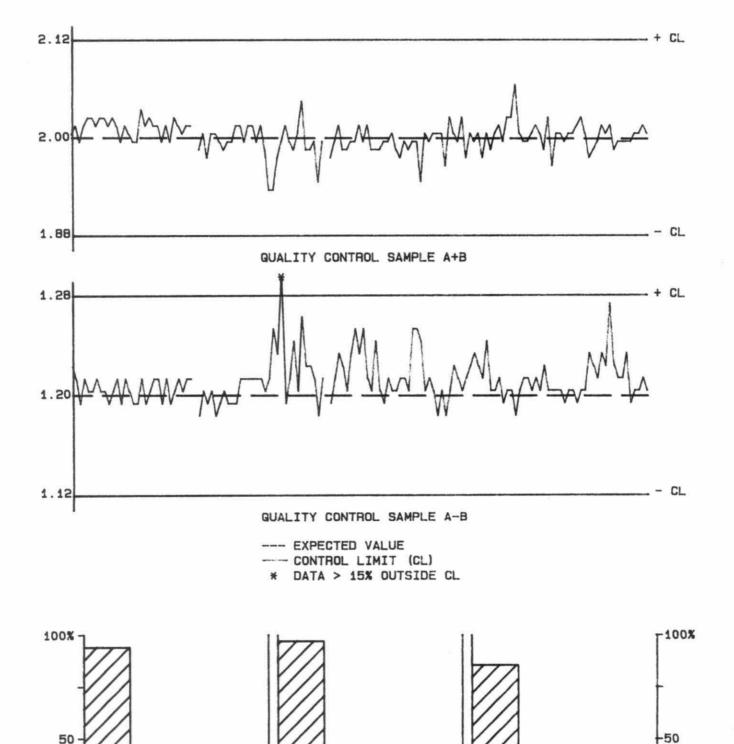
On any given day the calibration is accepted if the values obtained lie within the ranges:

1.88 to 2.12 for A+B 1.12 to 1.28 for A-B

DUPLICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Spa	an	s.d.	of var.(%)
	25	0.00 -	0.20	0.018	17.6
	57	0.20 -	0.50	0.011	3.3
	57	0.50 -	1.00	0.011	1.6
	33	1.00 -	2.00	0.020	1.4
	172	Overall	l	0.014	N/A

QUALITY CONTROL GRAPHS NITROGEN-NITRATE (MG/L AS N)

FROM: 02/01/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

20-50% F.S.

4-6% > 6%

0-2%

2-4%

50-100% F.S.

4-6%

> 6%

2-4%

0-2%

2-4%

4-6%

0-20% F.S.

> 6%

0-2%

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80

LIS Test Name Code: NNO3FR NNRICF Units : ug/Filter as N

Work Station Code : PRSEQ Unit Code : 361807
Method Code : 004AI0 Supervisor : M. Rawlings

Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and nylon filters from LoVol filter packs.

SAMPLING

Quantity Required: 1 filter

Container : Polyethylene bags

SAMPLE PREPARATION:

Filters are extracted with 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03N NaOH (nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as N.

Full scale conductivity : 30 uS/cm.

Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

-Ultrasonic bath; polystyrene tubes

-Automated modular continuous flow ion chromatographic system.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.25 ug/filter Detection Criterion (T): 0.5 ug/filter

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples.

MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified for analysis of Teflon and nylon filter extracts by developing the above filter extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

10/05/85 - Microcomputeer used for data reduction. Three additional calibration standards were set up.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

NITROGEN - NITRATE QUALITY CONTROL DATA FROM 04/01/85 TO 27/12/85

Lab: Precipitation

Analytical Range: 0.5 to 50.0 ug/Filter as N

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	I	159	40.0	40.0	0.0	0.32
ь	:	159	10.0	10.0	0.0	0.21
a+b	1	159	50.0	50.0	0.0	0.40
a-b	E	159	30.0	29.9	-0.1	0.38

s.d.(AB): Sw(within run): 0.27 S(between runs):

0.27 S/Sw: 1.01

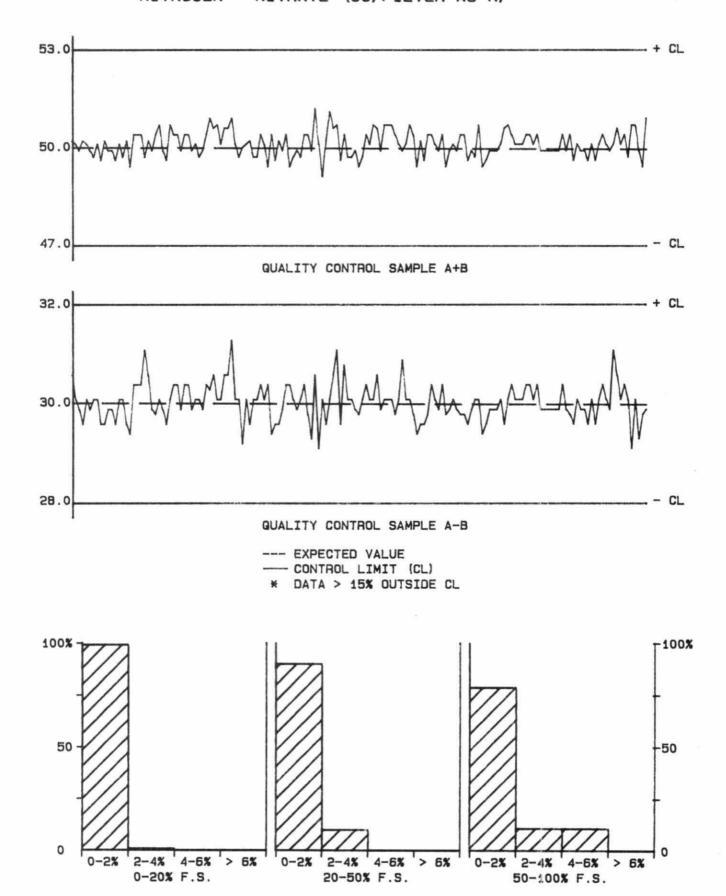
On any given day the calibration is accepted if the values obtained lie within the ranges:

47.0 to 53.0 for A+B 28.0 to 32.0 for A-B

DUPLICATES:	Number of Data Pairs	Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
	196	0.0 -	5.0	0.18	11.1
	55	5.0 -	10.0	0.34	4.6
	39	10.0 -	25.0	0.48	3.1
	24	25.0 -	50.0	0.86	2.4
	314	Overa	11	0.35	N/A

QUALITY CONTROL GRAPHS NITROGEN - NITRATE (UG/FILTER AS N)

FROM: 04/01/85 TO: 27/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 UG/FILTER AS N

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80

LIS Test Name Code: NNO3UR Units : ug/Filter as N

Work Station Code : PRLOV Unit Code : 361807

Method Code : 004AIC Supervisor : M. Rawlings

Sample Type/Matrix: W40 filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bag

SAMPLE PREPARATION:

Filters are extracted with 50.0mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as N.

Full scale conductivity : 30 uS/cm.

Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

- Ultrasonic bath; polyethylene tubes
- Automated modular continuous flow ion chromatographic system.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.5 ug/filter Detection Criterion (T): 1.2 ug/filter

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : 1 standard every 10 samples.

MODIFICATIONS:

01/08/81 - Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

10/05/85 - Microcomputer used for data reduction. Three additional calibration standards were set up.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

No data summary is available for period not covered in performance report.

NITROGEN - NITRATE QUALITY CONTROL DATA FROM 05/02/85 TO 12/12/85

Lab: Precipitation

Analytical Range: 1.2 to 100.0 ug/Filter as N

CHEADINI LONG CONTINUES	CAL	IBRAT	NOI	CONTROL
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		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	27	80.0	79.9	-0.1	0.67
b	:	27	20.0	20.0	0.0	0.43
a+b	:	27	100.0	99.9	-0.1	0.84
a-b	:	27	60.0	60.0	0.0	0.75

s.d.(AB): Sw(within run): 0.53 S(between runs): 0.56 S/Sw: 1.06

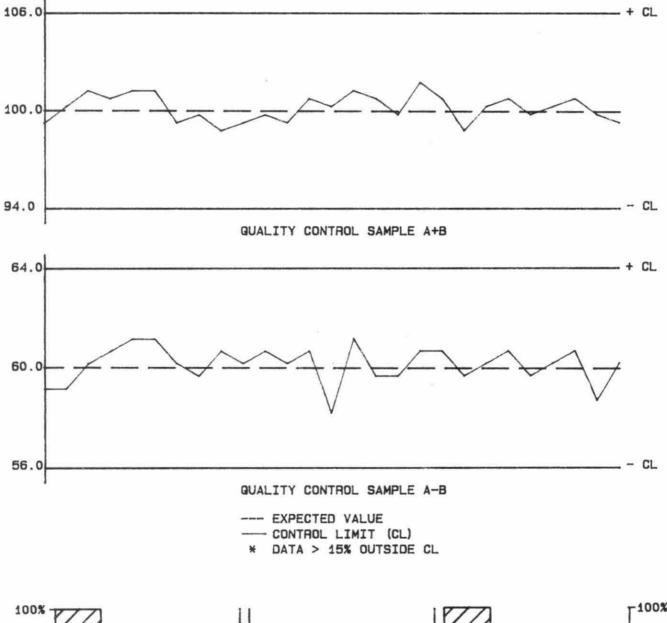
On any given day the calibration is accepted if the values obtained lie within the ranges:

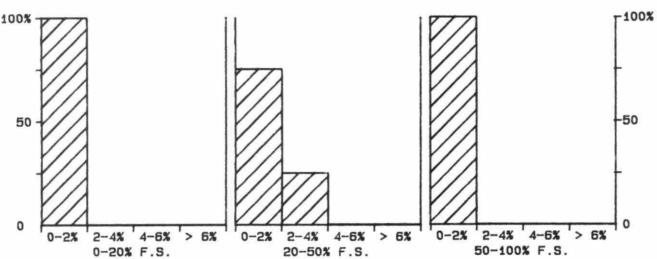
94.0 to 106.0 for A+B 56.0 to 64.0 for A-B

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	24	0.0 - 10.0	0.41	9.7
	5	10.0 - 25.0	0.82	4.4
	7	25.0 - 50.0	1.19	3.4
	2	50.0 - 100.0	0.56	0.8
	38	Overall	0.69	N/A

QUALITY CONTROL GRAPHS NITROGEN - NITRATE (UG/FILTER AS N)

FROM: 05/02/85 TO: 12/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 UG/FILTER AS N

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/04/78
LIS Test Name Code: NNOTUR Units : mg/L as N
Work Station Code: WFN03 Unit Code : 064807
Method Code : 002CC2 Supervisor : M. Rawlings

Sample Type/Matrix: Ministry of Health Water Samples

SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.5 at 20.0 mg/L as N level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 0.1

CALIBRATION:

BL plus 1 standard in duplicate

CONTROLS:

Calibration : 2 standards, eg, QCA Drift : BL plus 2 standards

NITROGEN-NITRATE+NITRITE QUALITY CONTROL DATA FROM 02/01/85 TO 27/12/85

Lab: Domestic Water

Analytical Range: 0.1 to 20.0 mg/L as N

CALIBRATION	CONTROL
-------------	---------

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	72	15.0	14.9	-0.1	0.17
ь	2	72	3.0	3.0	0.0	0.09
a+b	:	72	18.0	17.9	-0.1	0.23
a-b	:	72	12.0	11.9	-0.1	0.15

s.d.(AB): Sw(within run): 0.11 S(between runs): 0.14 S/Sw: 1.28

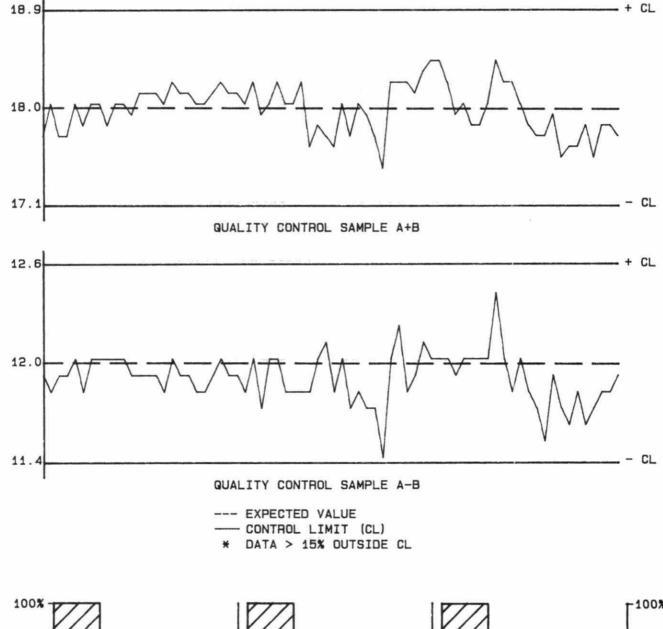
On any given day the calibration is accepted if the values obtained lie within the ranges:

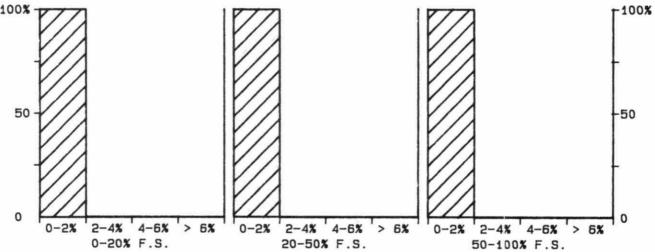
> 17.1 to 18.9 for A+B 11.4 to 12.6 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Sp		Mean(2)	Coefficient of var.(%)
	143	0.0 -	2.0	0.03	7.4
	32	2.0 -	5.0	0.07	2.1
	14	5.0 -	10.0	0.08	1.0
	10	10.0 -	20.0	0.07	0.5
	199	Overal	1	0.05	N/A

QUALITY CONTROL GRAPHS NITROGEN-NITRATE+NITRITE (MG/L AS N)

FROM: 02/01/85 TO: 27/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 20 MG/L AS N

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 13/06/78
Supervisor : F. Tomassini Units : ug/L as N

Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL

Container : Plastic (polystyrene)

SAMPLE PREPARATION:

Samples are filtered through 0.45u membrane filters.

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the filtrate of a sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and NK 1-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.4 at the 500 ug/L as N level. N.B. Ammonia plus ammonium is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 ml. delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 2

Detection Criterion (T): 9

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard every 10 samples

NITROGEN - NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 10/01/85 TO 19/12/85

Lab: Dorset

Analytical Range: 9 to 500 ug/L as N

CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	49	350	354	4	3.8
ь		49	125	128	3	2.8
a+b	:	49	475	482	7	5.6
a-b	:	49	225	226	1	3.8

s.d.(AB): Sw(within run): 2.7 S(between runs): 3.3 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

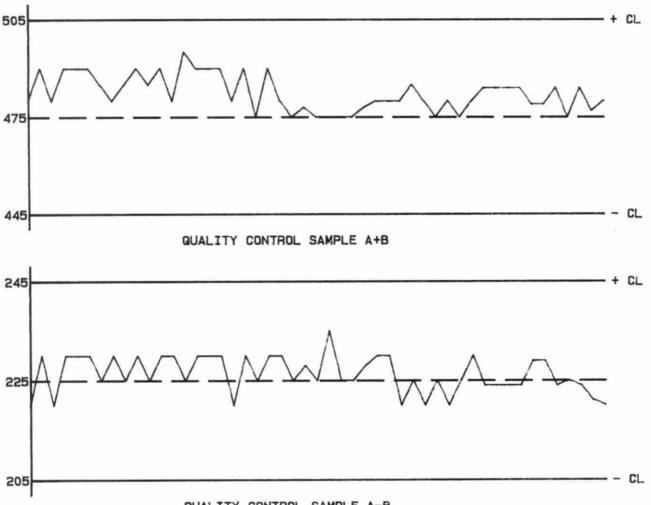
> 445 to 505 for A+B 205 to 245 for A-B

DUPLICATES	Number of	s	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Span		Span	s.d.	of var.(%)
	75	Ø	-	50	3.0	16.2
	25	50	-	100	3.5	4.6
	20	100	-	250	3.6	2.3
	7	250	-	500	4.2	1.3
	127	0	vera	11	3.3	N/A

OTHER CHECKS:	Number		Data	Standard(1) Deviation
	of Data		Me an	
Std. Cal.	:	49	336	70.7
Long Term Blank	:	49	Ø	0.7

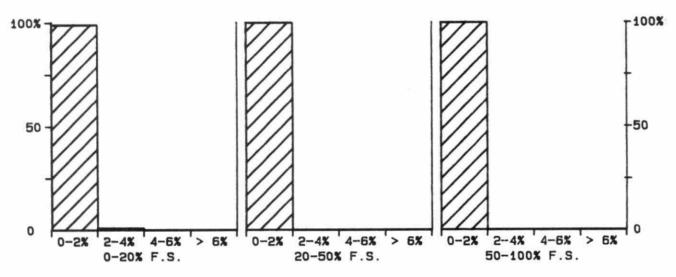
QUALITY CONTROL GRAPHS NITROGEN - NITRATE PLUS NITRITE (UG/L AS N)

FROM: 10/01/85 TO: 19/12/85



QUALITY CONTROL SAMPLE A-B

--- EXPECTED VALUE - CONTROL LIMIT (CL) * DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 UG/L AS N

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNOTFR Units : mg/L as N

Work Station Code : RNDNP Unit Code : 064807

Method Code : 102DC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and NK1-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.5 at the 5.00 mg/L as N level.

N.B. Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following moduules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.005

Detection Criterion (T): 0.06

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration: LTBL plus 3 standards

Drift : BL plus 1 standard

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

NITROGEN-NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

Lab: Rivers and Lakes Analytical Range: 0.06 to 5.00 mg/L as N

CAL	TRRAT	MOT	CON	TROL:
unu	IDKI	1014	CUIT	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	137	4.00	3.98	-0.02	0.039
b		137	1.00	1.00	0.00	0.017
a+b	:	137	5.00	4.98	-0.02	0.045
a-b	I	137	3.00	2.98	-0.02	0.040
c	:	137	1.00	1.00	0.00	0.017
d	:	137	0.50	0.50	-0.00	0.015
c +d	:	137	1.50	1.50	-0.00	0.030
c-d	:	137	0.50	0.50	0.00	0.013

s.d.(AB): Sw(within run): 0.028 S(between runs): 0.030 S/Sw: 1.06 s.d.(CD): Sw(within run): 0.009 S(between runs): 0.016 S/Sw: 1.74

On any given day the calibration is accepted if the values obtained lie within the ranges:

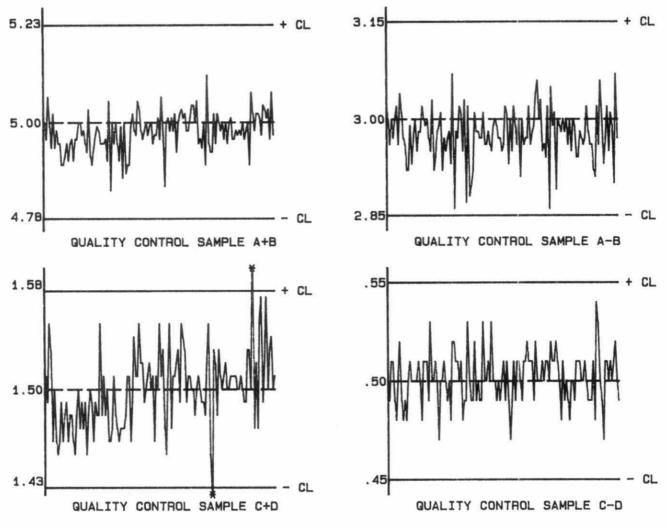
4.77 to 5.23 for A+B 2.85 to 3.15 for A-B 1.43 to 1.58 for C+D 0.45 to 0.55 for C-D

DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	144	0.00 - 0.20	0.020	28.9
	69	0.20 - 0.50	0.032	10.1
	41	0.50 - 1.00	0.031	4.3
	83	1.00 - 2.50	0.075	4.4
	33	2.50 - 5.00	0.079	2.2
	370	Overal1	0.047	N/A

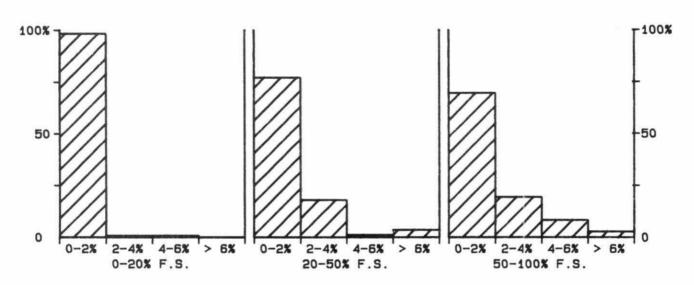
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	136	0.02	0.010

QUALITY CONTROL GRAPHS NITROGEN-NITRATE PLUS NITRITE (MG/L AS N)

FROM: 03/01/85 TO: 20/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS N

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/76
LIS Test Name Code: NNOTFR Units : mg/L as N
Work Station Code: SNO2NO3 Unit Code : 064807
Method Code: 102CC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl)ethylenediaminedihydrochloride. Approximate absorbance: 0.2 at 20.0 mg/L as N level. N.B. Nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.24

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: LTBL plus 4 standards
Drift: BL plus 2 standards

Interference: Nitrite standard (nitrate and nitrite at same concentration run separately: zero difference is expected) confirms effective operation of reduction step. Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective operation of ion exchange column.

MODIFICATIONS:

01/06/85 -Ion exchange column was removed and replaced by increasing in-line sample dilution to the point that the interference check could be retained and no loss in performance was observed.

NITROGEN - NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 03/01/85 TO 27/12/85

Lab: Sewage and Industrial Waste Analytical Range: 0.24 to 50.0 mg/L as N

CAL.	IBRAT:	MOI	CON	TROLI
~ 11	ADINITI	•	~~,	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	114	35.0	35.3	0.3	0.37
ь	:	114	15.4	15.7	0.3	0.27
a+b	:	114	50.4	51.0	0.6	0.55
a-b	:	114	19.6	19.7	0.1	0.35
С	:	114	15.40	15.66	0.26	0.213
d		114	4.20	4.33	0.13	0.089
c+d		114	19.60	19.99	0.39	0.265
c -d		114	11.20	11.33	0.13	0.190

s.d.(AB): Sw(within run): 0.25 S(between runs): 0.32 S/Sw: 1.31 s.d.(CD): Sw(within run): 0.134 S(between runs): 0.163 S/Sw: 1.21

On any given day the calibration is accepted if the values obtained lie within the ranges:

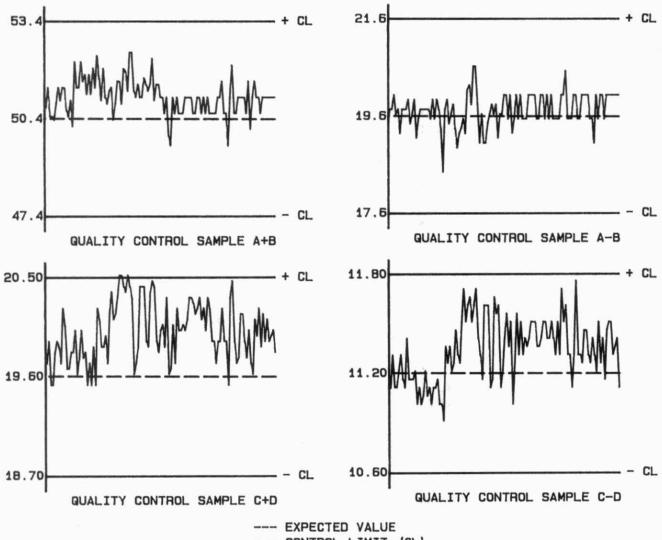
47.4 to 53.4 for A+B 17.6 to 21.6 for A-B 18.70 to 20.50 for C+D 10.60 to 11.80 for C-D

DUPLICATES	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn	Span	s.d.	of var.(%)
	235	0.00 -	2.00	0.080	19.6
	36	2.00 -	5.00	0.100	2.8
	31	5.00 -	10.00	0.115	1.7
	27	10.0 -	20.0	0.24	1.6
	6	20.0 -	50.0	0.43	1.5
	335	Over	al1 .	0.12	N/A

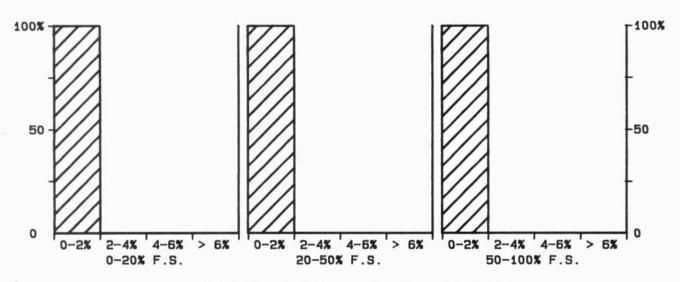
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
		~~~~		
Std. Cal	:	112	551	78.4
N03-N02	:	105	0.10	0.188
NO3-Ca/Mg	:	104	0.01	0.286
Long Term Blank		65	0.05	0.000

# QUALITY CONTROL GRAPHS NITROGEN - NITRATE PLUS NITRITE (MG/L AS N)

FROM: 03/01/85 TO: 27/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS N

#### *** NITROGEN - NITRITE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNO2FR Units : mg/L as N
Work Station Code: RNDNP Unit Code : 064807

Method Code : 102DC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

## ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and NK1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance: 0.6 at the 0.25 mg/L as N level.

N.B. Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.0005

Detection Criterion (T): 0.003

# CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration: LTBL plus 3 standards, e.g., QCA

Drift : BL plus 1 standard

### MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

# NITROGEN-NITRITE QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

Lab: Rivers and Lakes Analytical Range: 0.003 to 0.250 mg/L as N

CALIBRO	101TE	CONTROL				
		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	138	0.200	0.198	-0.002	0.0020
ь		138	0.050	0.050	-0.000	0.0011
a+b	:	138	0.250	0.248	-0.002	0.0023
a-b	1	138	0.150	0.149	-0.001	0.0022
c	:	138	0.050	0.050	-0.000	0.0011
d		137	0.025	0.025	-0.000	0.0010
c+d	:	137	0.075	0.074	-0.001	0.0018
c-d	:	137	0.025	0.025	-0.000	0.0010

s.d.(AB): Sw(within run): 0.0016 S(between runs): 0.0016 S/Sw: 1.04 s.d.(CD): Sw(within run): 0.0007 S(between runs): 0.0011 S/Sw: 1.49

On any given day the calibration is accepted if the values obtained lie within the ranges:

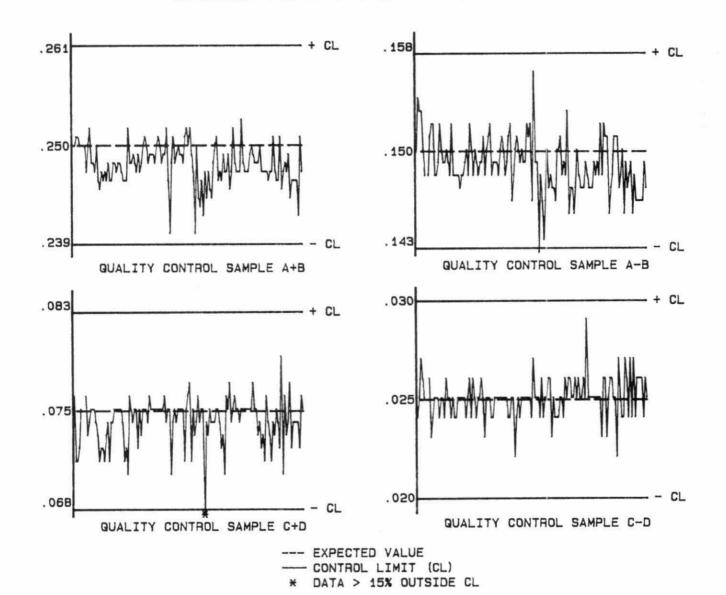
0.239 to 0.261 for A+B 0.142 to 0.157 for A-B 0.067 to 0.082 for C+D 0.020 to 0.030 for C-D

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	202	0.000 - 0.010	0.0010	24.1
	63	0.010 - 0.020	0.0018	12.9
	92	0.020 - 0.100	0.0037	8.4
	16	0.100 - 0.250	0.0068	4.1
	373	Overall	0.0026	N/A

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Elank	135	0.002	0.0008

# QUALITY CONTROL GRAPHS NITROGEN-NITRITE (MG/L AS N)

FROM: 03/01/85 TO: 20/12/85



50 50 50

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .25 Mg/L AS N

20-50% F.S.

4-6%

> 6%

0-2%

2-4%

50-100% F.S.

2-4%

0-2%

> 6%

4-6%

2-4%

0-20% F.S.

#### *** NITROGEN - NITRITE ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/76
LIS Test Name Code: NNO2FR Units : mg/L as N
Work Station Code: SNO2NO3 Unit Code : 064807
Method Code : 102CC2 Supervisor : P. Campbell

Method Code : 102CC2 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

**Effluents** 

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and N(1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance :0.4 at the 2.0 mg/L as N level. N.B. Nitrate plus nitrite is determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

# REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.005

Detection Criterion (T): 0.009

#### CALIBRATION:

BL plus 2 standards

# CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL plus 2 standards

# NITROGEN - NITRITE QUALITY CONTROL DATA FROM 03/01/85 TO 27/12/85

Lab: Sewage and Industrial Waste Analytical Range: 0.009 to 2.00 mg/L as N

CAL I	BRAT	ION	CONTROL:
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		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
c	:	112	1.400	1.398	-0.002	0.0271
1000	:	112	0.700	0.692	-0.008	0.0239
c+d	:	112	2.100	2.090	-0.010	0.0494
c-d		112	0.700	0.707	0.007	0.0132

s.d.(CD): Sw(within run): 0.0093 S(between runs): 0.0256 S/Sw: 2.74

On any given day the calibration is accepted if the values obtained lie within the ranges:

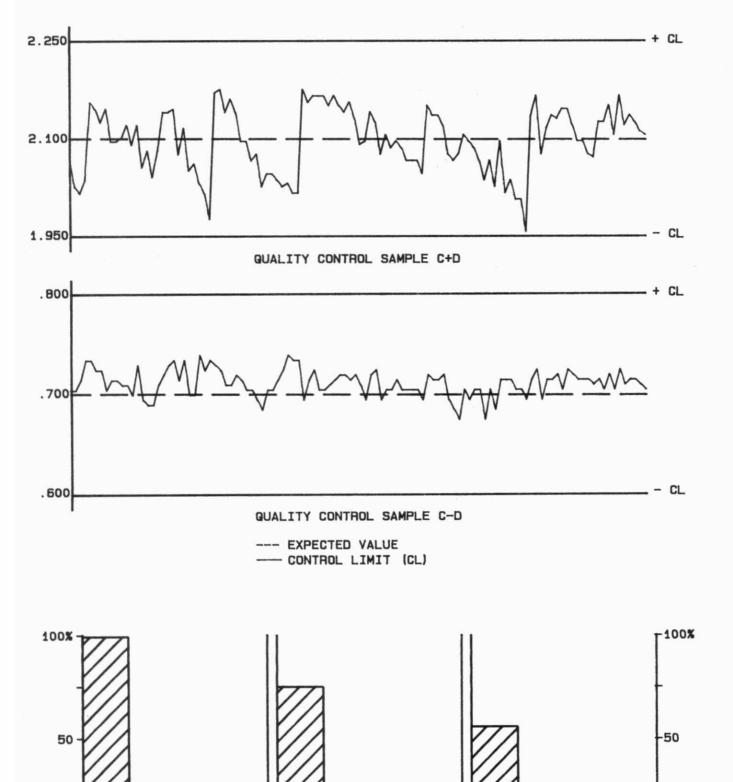
1.950 to 2.250 for C+D 0.600 to 0.800 for C-D

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	283	0.000 - 0.200	0.0031	17.0
	34	0.20 - 1.00	0.025	4.9
	8	1.00 - 2.00	0.043	3.1
	325	Overal1	0.011	N/A

OTHER CHECKS:	Number of Data		Data Mean	Standard(1) Deviation
Std. Cal	:	112	443	32.9
Long Term Blank		65	0.005	0.0006

# QUALITY CONTROL GRAPHS NITROGEN - NITRITE (MG/L AS N)

FROM: 03/01/85 TO: 27/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 MG/L AS N

20-50% F.S.

> 6%

50-100% F.S.

2-4% 4-6%

0-20% F.S.

#### *** NITROGEN-TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/79
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code : RTNP Unit Code : 064807
Method Code : 004AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance: 0.5 at the 2.0 mg/L as N level. N.B. Total phosphorus is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system plus 1 module:37 C bath(7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

-Data capture, reduction, and processing via a multi-stage microcomputer system REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.06

## CALIBRATION:

BL plus 3 undigested standards

### CONTROLS:

Calibration: LTBL plus 2 undigested standards, eg, QCA

Recovery : 3 digested BL plus 3 digested standards in duplicate, eg,R1

Drift : BL plus 1 undigested standard

# MODIFICATIONS:

15/08/83 - Microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic.

## NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

## NITROGEN-TOTAL KJELDAHL QUALITY CONTROL DATA FROM 03/01/85 TO 23/12/85

Lab: Rivers and Lakes

Analytical Range: 0.06 to 2.00 mg/L as N

CALIBRATION CONTROL:	1
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		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	ı	157	1.50	1.49	-0.01	0.017
b	:	157	0.50	0.51	0.01	0.015
a+b	:	157	2.00	2.00	0.00	0.027
ab	:	157	1.00	0.98	-0.02	0.019

s.d.(AB): Sw(within run): 0.013 S(between runs): 0.016 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B 0.94 to 1.06 for A-B

Number Ex	xpected Av.	.Conc. Stand	Standard(1)
f Data Co	oncn Meas	sured Devia	tion
155	1.40	1.37 0.0	<b>039</b>
156	0.84	0.83 0.	022
156	0.28	0.28 0.	011
1	f Data Co  155 156	f Data Concn Meas 	F Data Concn Measured Devia 155 1.40 1.37 0.0 156 0.84 0.83 0.0

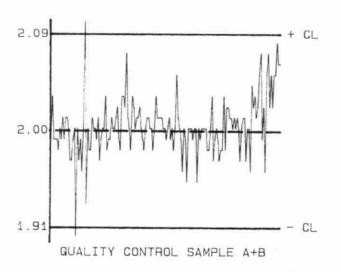
DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	55	0.00 - 0.	20 0.019	14.2
	216	0.20 - 0.	50 0.034	10.4
	97	0.50 - 1.	00 0.055	8.6
	13	1.00 - 2.	00 0.164	12.7
	381	Overall	0.049	N/A

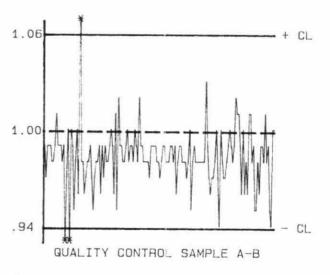
#### DETECTION CRITERION: 0.06

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	154	0.01	0.009
Digested Blank	:	155	0.02	0.008

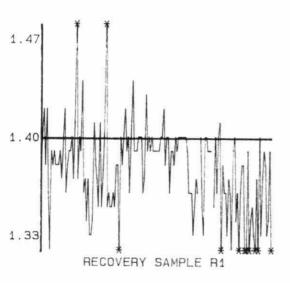
### QUALITY CONTROL GRAPHS NITROGEN-TOTAL KJELDAHL (MG/L AS N)

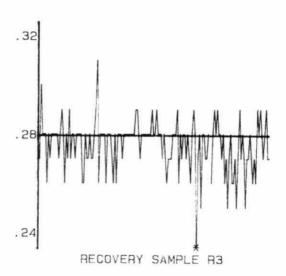
FROM: 03/01/85 TO: 23/12/85



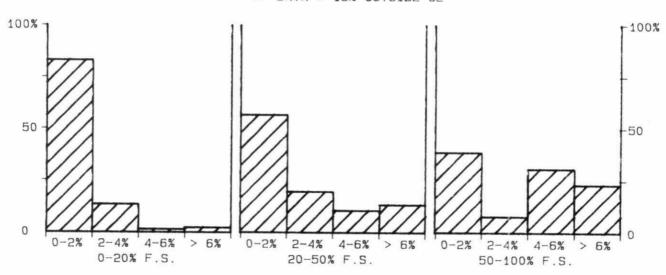


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

#### *** NITROGEN - TOTAL KJELDAHL ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/79
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code: STKNP Unit Code : 064807
Method Code : 504BC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Effluents,
Leachates

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance: 0.8 at the 10.0 mg/L as N level. N.B. Total phosphorus is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system plus 1 module:37 C bath(7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.19**

#### CALIBRATION:

BL plus 2 undigested standard

#### CONTROLS:

Calibration : LTBL plus 4 undigested standards, eg, QCA

Recovery : 2 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL plus 2 undigested standard

#### MODIFICATIONS:

01/10/85 -High range added, full scale changed from 10 to 25 mg/L as N. New calibration controls added.

#### NOTES:

**Minimum dilution is 50% (i.e. factor of two). Therefore actual minimum W and T values are 0.1 and 0.4 respectively.

#### NITROGEN - TOTAL KJELDAHL QUALITY CONTROL DATA FROM 03/01/85 TO 31/12/85

Lab: Sewage and Industrial Waste Analytical Range: 0.19 to 25.0 mg/L as N

	TODAT		COMPOS	
L.PIL.	IBKHI	11114	CONTROL	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	40	17.5	17.5	0.0	0.32
ь	1	40	7.0	7.2	0.2	0.23
a+b	:	40	24.5	24.7	0.2	0.52
a-b		40	10.5	10.4	-0.1	0.19
c	:	165	7.00	7.11	0.11	0.120
d	:	165	1.40	1.47	0.07	0.054
c+d	1	165	8.40	8.58	0.18	0.152
c-d		165	5.60	5.64	0.04	0.107

s.d.(AB): Sw(within run): 0.13 S(between runs): 0.28 S/Sw: 2.07 s.d.(CD): Sw(within run): 0.076 S(between runs): 0.093 S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.4 to 25.6 for A+B 9.8 to 11.2 for A-B 7.95 to 8.85 for C+D 5.30 to 5.90 for C-D

RECOVER IES:		Number	Expected	Av.Conc.	Standard(1)	
		of Data	Concn	Measured	Deviation	
r 1	:	130	17.5	17.3	0.51	
r2	:	151	7.00	6.93	0.192	
r3	:	151	3.50	3.51	0.127	

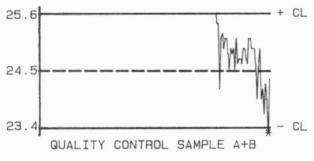
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	249	0.00 - 1.00	0.063	17.5
	34	1.00 - 2.00	0.061	4.4
	46	2.00 - 5.00	0.149	4.2
	26	5.0 - 10.0	0.19	2.8
	8	10.0 - 25.0	0.37	2.4
	363	Overal1	0.11	N/A

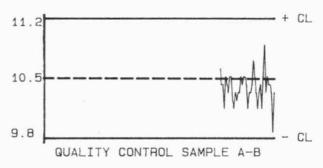
### DETECTION CRITERION: 0.19

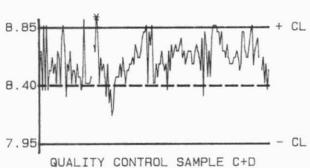
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal		143	181	34.8
Long Term Blank	:	155	0.05	0.010
Digested Blank	:	132	0.05	0.013

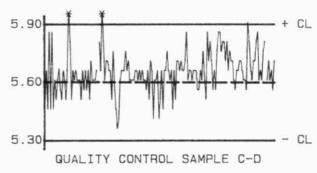
## QUALITY CONTROL GRAPHS NITROGEN - TOTAL KJELDAHL (MG/L AS N)

FROM: 03/01/85 TO: 31/12/85

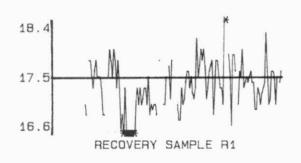


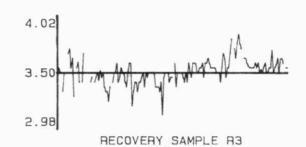




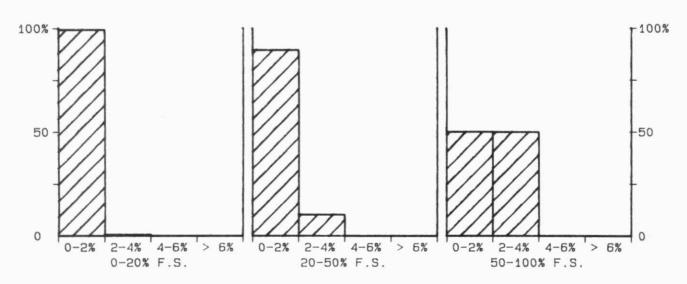


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 MG/L AS N

#### 157 *** OXYGEN-BIOCHEMICAL DEMAND ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: BOD5 Units : mg/L as O
Work Station Code : R5DBOD Unit Code : 064808
Method Code : 101A12 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

#### SAMPLING:

Quantity Required: 400 mL

Container : Glass or plastic

#### SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

#### ANALYTICAL PROCEDURE:

Using dissolved oxygen (DO) analyses, samples are measured for oxygen depletion after a five day period (BOD5) of storage in the dark at 20 C. If necessary dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples calculation of an appropriate seed correction is required.

#### INSTRUMENTATION:

- Weston and Stack Oxygen analyzer plus DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.
- Titration equipment for WinKler analysis of dissolved oxygen
- Incubator(19-21 C); BOD bottles(300mL)

#### REPORTING

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.67

#### CALIBRATION DO):

Blank is a sulphite solution (negligible DO) and the standard is air-saturated distilled water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

#### CONTROLS:

Calibration(DO): 2 "solutions" of distilled water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are analyzed with the DO probe and by the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO probe.

Recovery(BOD5): 2 BL plus 3 standards, eg, R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift(DO) : Air saturated distilled water after every 20 samples.

#### MODIFICATIONS

01/07/82 -Quality control program for DO was expanded, and the use of standard 300 mL BOD bottles was restored.

01/09/85 -BOD test transferred to Sewage/Industrial laboratory to provide consistent QC and improve productivity. No data summary is available for period not covered in performance report.

## OXYGEN -BIOCHEMICAL DEMAND QUALITY CONTROL DATA FROM 18/01/85 TO 28/08/85

Lab: Rivers and Lakes

Analytical Range: 0.67 to 20.0 mg/L as 0

CAL.	TRRAT	MOL	CON	TROL:	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	2	59	0.00	0.19	0.19	0.142
b	:	59	0.00	0.17	0.17	0.124

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.50 to 0.50

RECOVERIES		Number of Data	Expected Concn	Av.Conc. Measured	Standard(1) Deviation	
r1 r2	:	31 31	4.34 2.17	4.45 2.38	0.397 0.312	
DUPLICATES:	Number Data P	100000	Sample Concn Span	Mean(2)	Coefficient of var.(%)	

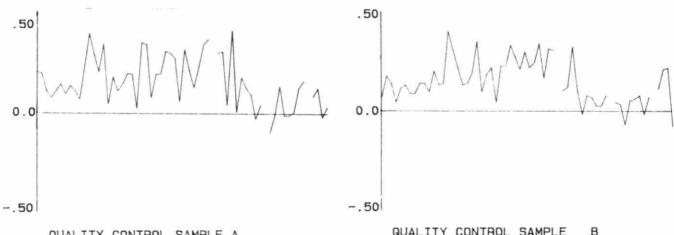
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%
	70	0.00 - 2.00	0.222	22.7
	12	2.00 - 10.00	0.164	4.7
	Ø	10.0 - 20.0	N/A	N/A
	82	Overal1	0.21	N/A

#### DETECTION CRITERION: 0.67

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
5 day BOD Blank	29	0.17	0.122

### QUALITY CONTROL GRAPHS OXYGEN -BIOCHEMICAL DEMAND (MG/L AS 0)

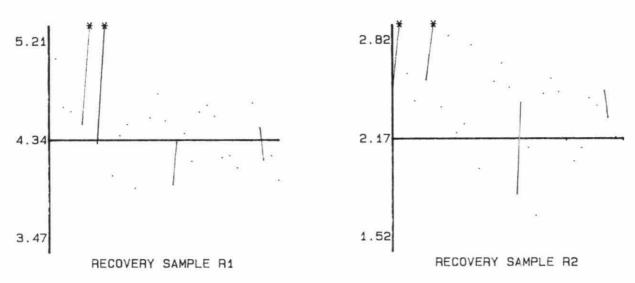
FROM: 18/01/85 TO: 28/08/85



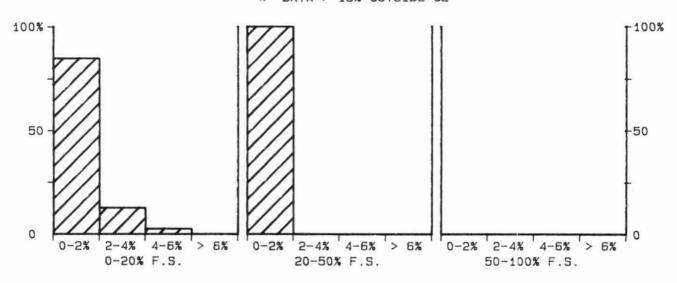
QUALITY CONTROL SAMPLE A

QUALITY CONTROL SAMPLE B

--- EXPECTED VALUE --- CONTROL LIMIT (CL)



* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS 0

#### *** OXYGEN - BIOCHEMICAL DEMAND ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: BOD5 Units : mg/L as O
Work Station Code : SBBOD5 Unit Code : 064808
Method Code : 101AI2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Effluents, Domestic Waters,
Leachates

#### SAMPLING:

Quantity Required: 400 mL

Container : Glass or plastic

#### SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

#### ANALYTICAL PROCEDURE:

Using dissolved oxygen (DO) analyses, samples are measured for oxygen depletion after a five day period (BOD5) of storage in the dark at 20C. If necessary dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples calculation of an appropriate seed correction is required.

#### INSTRUMENTATION:

- Weston and Stack Oxygen analyzer plus DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.
- Titration equipment for Winkler analysis of dissolved oxygen
- Incubator(19-21 C); BOD bottles (300mL)

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 1.0

#### CALIBRATION DO >:

Blank is a sulphite solution(negligible DO) and the standard is air-saturated distilled, deionized water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

#### CONTROLS:

Calibration(DO): 2 "solutions" of distilled water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are analyzed with the DO probe and by the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO probe.

Recovery(BOD5): 2 BL plus 3 standards, eg, R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift(DO) : Air saturated distilled water after every 24 samples.

#### MODIFICATIONS

01/05/81 -Quality control program for DO was expanded, and the use of standard 300 mL BOD bottles was restored.

25/06/84 -Digital burette (readability to 0.01mL) replaced glass burette.

### OXYGEN DEMAND - BIOCHEMICAL QUALITY CONTROL DATA FROM 02/01/85 TO 19/12/85

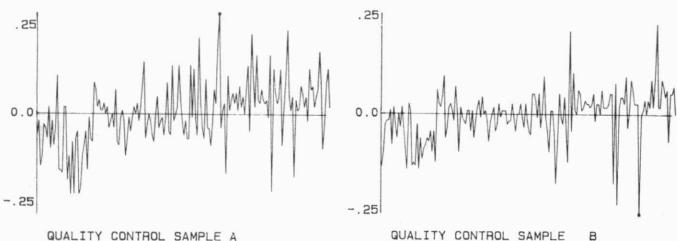
Lab: S	ewage	and Industri	al Waste	Analytical	Range:	1.0	to 4	100	mg/L	as	L
CALIBR	ATION	CONTROL									
		Number	Expected	Av.Concn	Av.	Bias		Sta	ndard	(1)	
		of Data	Concn	Measured				De	viati	on	
											è
a	:	175	0.00	0.00	e	.00			0.092		
		175	0 00	-0 01	-0	1.01			0.074		

A and B

On any given day th lie within the rang			s accepte		s obtained for
RECOVER IES:	Number	E	xpected	Av.Conc.	Standard(1)
11200121120	of Data		oncn	Measured	Deviation
r1 :	80		2.17	2.11	0.128
r2 :	79		4.34	4.21	0.157
r3 !	80		6.52	6.35	0.159
DUPLICATES: Number	er of	Sam	nple	Mean(2)	Coefficient
Data	Pairs	Concn	Span	s.d.	of var.(%)
	9	0.0 -	5.0	0.33	13.0
5	4	5 -	- 20	1.4	11.0
3:	9	20 -	50	3.3	9.5
4	1	50 -	100	5.2	6.9
5:	3	100 -	400	12.5	7.3
17	2	Ove	rall	7.9	N/A
DETECTION CRITERIO	N: 1.0				
OTHER CHECKS:		Number		Data	Standard(1)
		of Data	k.	Mean	Deviation
			-		
5 day DDW Blank	:	89		0.16	0.090
5 day BOD Blank	:	88		0.15	0.109

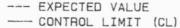
### QUALITY CONTROL GRAPHS OXYGEN DEMAND - BIOCHEMICAL (MG/L AS 0)

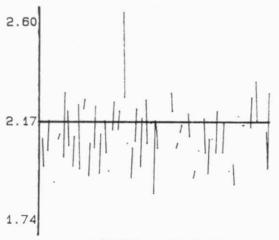
FROM: 02/01/85 TO: 19/12/85



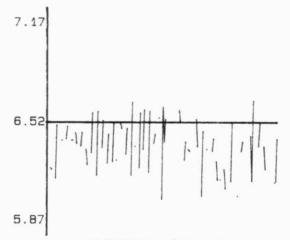
QUALITY CONTROL SAMPLE A

QUALITY CONTROL SAMPLE



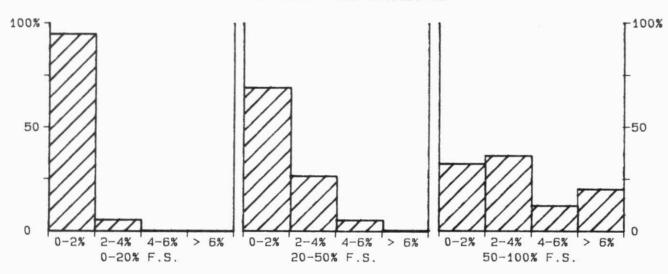


RECOVERY SAMPLE R1



RECOVERY SAMPLE R3





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 400 MG/L AS 0

#### *** DXYGEN - CHEMICAL DEMAND ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/07/82
LIS Test Name Code: COD Units : mg/L as 0
Work Station Code: RCOD Unit Code : 064808

Method Code : 002AC0 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPLING:

Quantity Required: 25 mL Container : Glass

#### ANALYTICAL PROCEDURE:

Samples(10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the 500 mg/L level.

#### INSTRUMENTION:

Culture tubes with Teflon closures; mechanical-convection oven -Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 1

Detection Criterion (T): 2

#### CALIBRATION:

2 digested BL plus 3 digested standards

#### CONTROLS:

Calibration: 2 digested standards, eg, QCA
Recovery: 2 digested standards, eg, R1
Drift: Undigested BL: 1 per 9 samples

Interference: Digested standard (40 mg/L as O) spiked with 50 mg/L C1 confirms suppression of chloride interference.

#### MODIFICATIONS:

30/06/82- Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request. NOTES:

-In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.

-Chemical oxygen demand analyses for Rivers and Lakes' samples are set up in the latter laboratory, but completed in the Sewage/Industrial laboratory. Thus the following performance data report only refers to duplicate results. Please consult the Sewage/Industrial report for the remaining QC data.

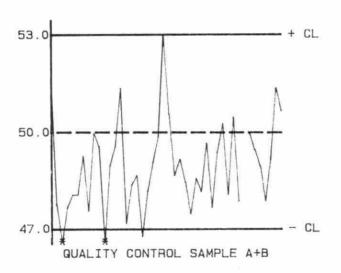
#### OXYGEN - CHEMICAL DEMAND QUALITY CONTROL DATA FROM 10/01/85 TO 13/12/85

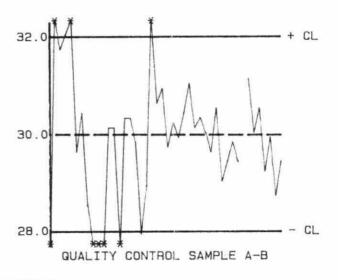
0

Lab: Rivers a	nd LaKes		Analytical	Range: 2	to 100 mg/L as
COL IDDATION C	ONTEROI I				
CALIBRATION C	Number	Expected	Av.Concn	Av.Bias	Standard(1)
	of Data	Concn	Measured	1101014	Deviation
	OT Data		reasured		
	43	40.0	39.3	-0.7	1.02
a : h :	43	10.0	9.6	-0.4	1.33
	43	50.0	48.9	-1.1	1.52
a+b :	43	30.0	29.7	-0.3	1.81
a-b :	43	30.0	20.1	-0.5	
s.d.(AB): Sw	(within run)	1.28	S(between r	uns): 1.19	9 S/Sw: 0.93
On any given	day the cali	bration is	accepted if	the values of	tained lie within
the ranges:	,				
tile l'aliges		47.0 t	o 53.0 for	A+B	
		28.0 t	o 32.0 for	A-B	
RECOVER IES:	Numb	er Exp	ected A	v.Conc.	Standard(1)
NEGO I EN I E	of Da		cn Me	asured I	Deviation
r1	: 41		40.0	36.3	3.06
re			10.0	8.7	1.97
DUPL ICATES:	Number of	Samp1	e Me	an(2) C	pefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	13	ø -	10 0.	5	7.8
	60	10 -	25 1.	6	9.5
	17	25 -	50 3.	3	10.4
	4	50 -	100 0.	3	0.6
	94	Overa	11 1.	9	N/A
DETECTION CRI	TERION: 2				
OTHER CHECKS		Number	Data	St	andard(1)
OTHER CHECKS		of Data	Mean		eviation
		OT Data			
Std Cal		21	484.1		80.2
Chloride I	1 2	21	39.6		1.41
Digested E		41	3.8		2.34
Diaested E	A SUIT		0.0		

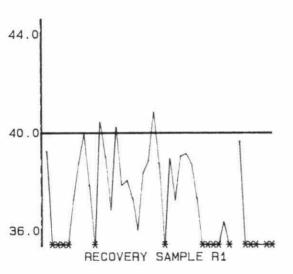
# QUALITY CONTROL GRAPHS OXYGEN - CHEMICAL DEMAND (MG/L AS 0)

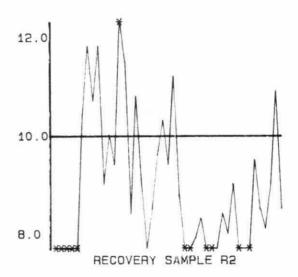
FROM: 10/01/85 TO: 13/12/85



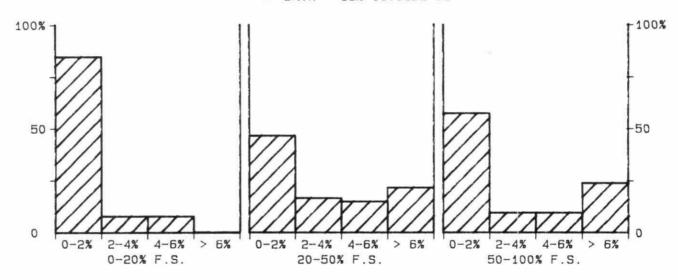


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 50 MG/L AS 0

*** OXYGEN - CHEMICAL DEMAND ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/07/82 LIS Test Name Code: COD Units : mg/L as O Work Station Code: SBCOD Unit Code : 064808

Method Code : 002AC0 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachates,

### Effluents

SAMPLING:

Quantity Required: 25 mL Container : Glass

#### ANALYTICAL PROCEDURE:

Samples(10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the 500 mg/L level.

#### INSTRUMENTION:

Culture tubes with Teflon closures; mechanical-convection oven -Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

#### REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 1

Detection Criterion (T): 13

#### CCALIBRATION:

2 digested BL plus 4 digested standards

#### CONTROLS:

Calibration: 2 digested standards, eg, QCA
Recovery: 2 digested standards, eg, R1
1 digested blank per 9 samples

Drift : 1 undigested BL per 9 samples

Interference: Digested standard (50 mg/L as 0) spiked to 900 mg/L C1 confirms suppression of chloride interference.

#### MODIFICATIONS:

30/06/82- Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request.

#### NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis are scheduled for completion within the week.

## OXYGEN DEMAND - CHEMICAL QUALITY CONTROL DATA FROM 09/01/85 TO 17/12/85

Lab: Sewage and Industrial Waste Analytical Range: 13 to 500 mg/L as O

CAL.	IBRAT	MOL	COM	TROL :
	ADICITI	1011		11/02 -

		Number	Number Expected		Av.Bias	Standard(1)	
		of Data	Concn	Measured		Deviation	
a	:	57	400	397	-3	13.1	
ь	:	57	100	101	1	6.8	
a+b	:	57	500	498	-2	18.3	
a-b	:	57	300	296	-4	10.2	

s.d.(AB): Sw(within run): 7.2 S(between runs): 10.4 S/Sw: 1.45

On any given day the calibration is accepted if the values obtained lie within the ranges:

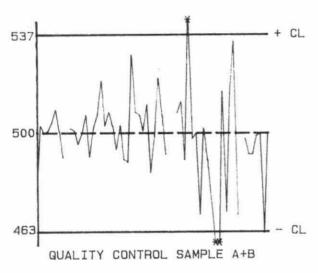
463 to 537 for A+B 275 to 325 for A-B

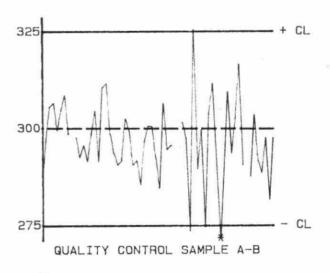
RECOVER IES:		Number of Data		Exp	ected	Av.Conc. Measured	Standard(1) Deviation
r1	:	57		4	100	387	10.9
r2	:	55		1	100	98	5.9
DUPLICATES:	Numbe	r of	S	a.m.p.1	e	Mean(2)	Coefficient
	Data	Pairs	Con	cn S	Span	s.d.	of var.(%)
	26	3	Ø	-	50	4.4	33.8
	4	1	50	-	100	2.0	2.8
	ε	3	100	-	250	15.1	9.2
	4	1	250	-	500	6.9	2.1
	36	6	0	vera	11	8.2	N/A
DETECTION CRI	TER ION	13					983

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
		,		
Std. Cal	:	50	246	17.1
Chloride Check	1	56	57	7.9
Digested Blank	:	58	41	9.8

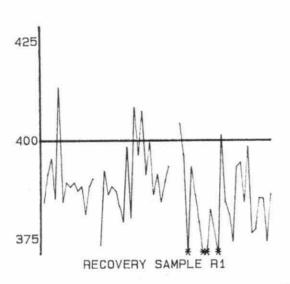
## QUALITY CONTROL GRAPHS OXYGEN DEMAND - CHEMICAL (MG/L AS 0)

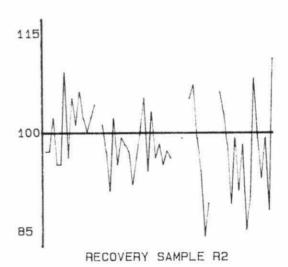
FROM: 09/01/85 TO: 17/12/85



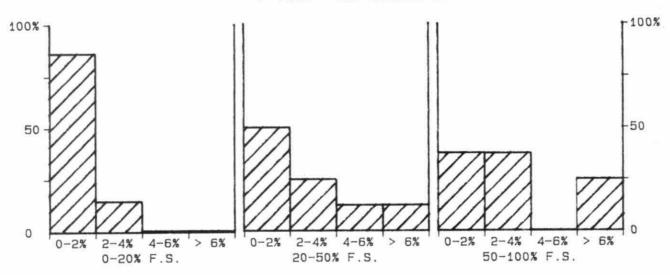


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS 0

#### *** PH ***

IDENTIFICATION:

Method Introduced: Before '60 : Domestic Water Laboratory Units : Dimensionless

: Nil

LIS Test Name Code: PH Unit Code

Work Station Code : WPC Supervisor : M. Rawlings Method Code : 001AI1

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

: Glass or plastic Container

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample at room temperature. Stirring rate, and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

pH meter, stirrer, glass electrode

REPORTING:

Maximum Significant Figures: 3

Detection Criterion (T): N/A Minimum Increment (W): 0.01

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

CONTROLS:

Calibration : 2 buffers

PH QUALITY CONTROL DATA FROM 03/01/85 TO 30/12/85

Lab: Domestic Water

Analytical Range: 0.00 to 14.00

CAL	IBRAT	MOL	CON	TROL

			Number	Expected	Av.Concn	Av.Bias	Standard(1)
			of Data	Concn	Measured		Deviation
	a	1	170	8.50	8.53	0.03	0.033
	b	1	170	6.20	6.19	-0.01	0.024
a	a+b	1	170	14.70	14.72	0.02	0.047
	a-b	:	170	2.30	2.33	0.03	0.035

s.d.(AB): Sw(within run): 0.025 S(between runs): 0.029 S/Sw: 1.17

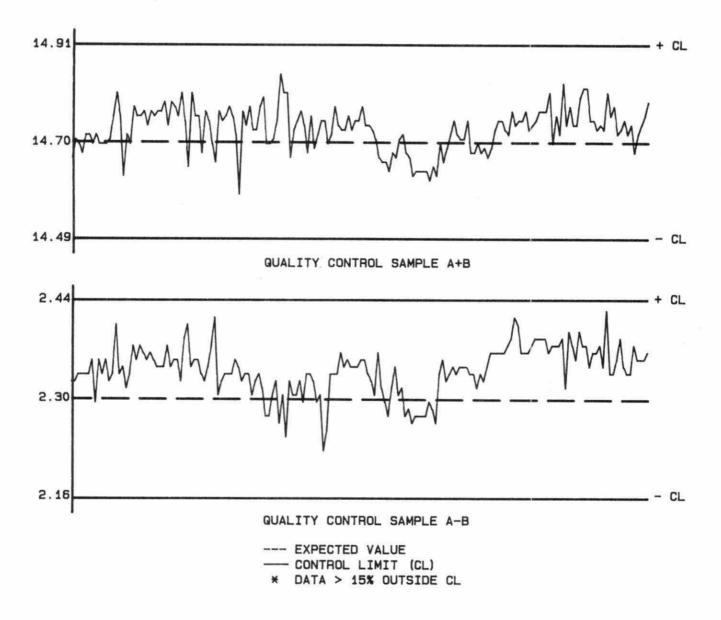
On any given day the calibration is accepted if the values obtained lie within the ranges:

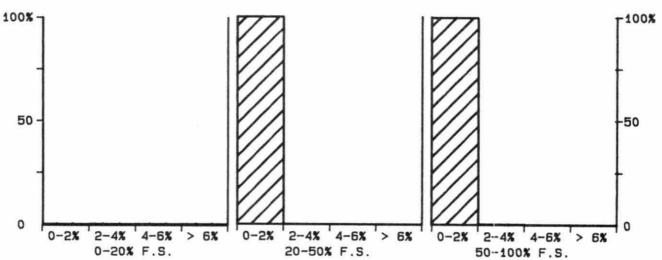
14.49 to 14.91 for A+B 2.16 to 2.44 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)	
	7	0.00 - 6.00	0.019	0.3	
	27	6.00 - 7.00	0.034	0.5	
	260	7.00 - 8.00	0.039	0.5	
	90	8.00 - 9.00	0.030	0.3	
	4	9.00 - 14.00	0.033	0.3	
	388	Overall	0.036	N/A	

## QUALITY CONTROL GRAPHS

FROM: 03/01/85 TO: 30/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### *** PH ***

#### IDENTIFICATION:

Laboratory

: Dorset

Method Introduced: 01/06/76

Supervisor

: F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipation

#### SAMPLING:

Quantity Required: 250 mL

Container

: Polyethylene bottle filled to the brim; screw caps with

cone-shaped liners

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (100 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

N.B. Two performance reports follow: in the first, Alkalinity (Gran) was performed simultanelusly, the second was a stand-alone pH work station.

#### INSTRUMENTATION

Semi-automated modular titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, eg, QCA
Drift: 2 standard buffers -4 times daily

PH QUALITY CONTROL DATA FROM 03/01/85 TO 24/12/85

Lab: Dorset

Analytical Range: 0.00 to 14.00

CAL	BRAT	HOL	CONTROL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		211	6.86	6.88	0.02	0.010
ь	I	211	4.00	4.00	-0.00	0.014
a+b		211	10.86	10.87	0.01	0.014
a-b	:	211	2.86	2.88	0.02	0.020

s.d.(AB): Sw(within run): 0.014 S(between runs): 0.012 S/Sw: 0.86

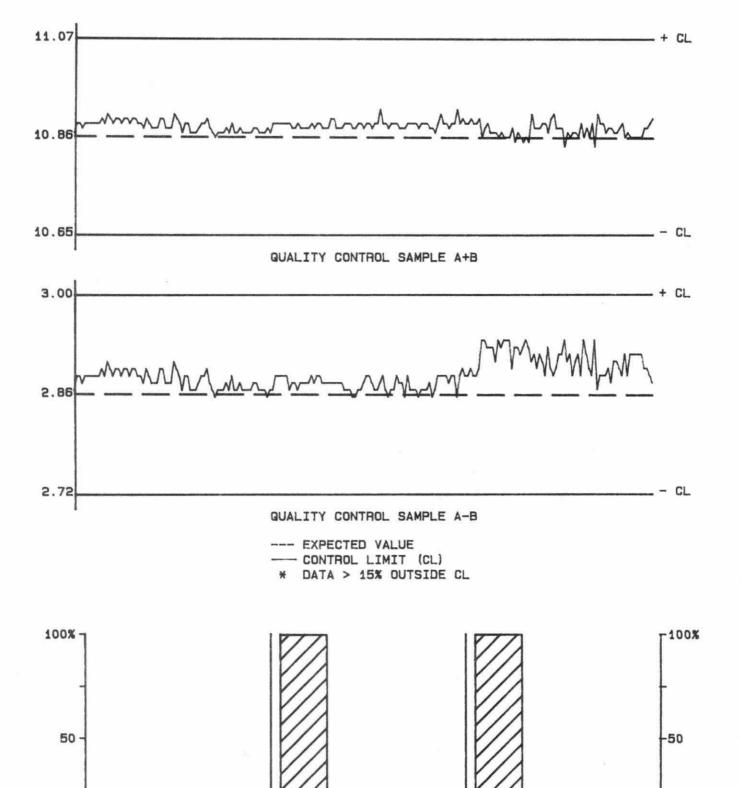
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 to 11.07 for A+B 2.72 to 3.00 for A-B

DUPL ICATES:	Number of Data Pairs	Sampl Concn S		Mean(2)	Coefficient of var.(%)
	Ø	0.00 -	4.00	N/A	N/A
	109	4.00 -	5.50	0.041	0.8
	202	5.50 -	7.00	0.040	0.6
	4	7.00 -	8.50	0.019	0.2
	Ø	8.50 -	14.00	N/A	N/A
	315	Overa	11	0.040	N/A

# QUALITY CONTROL GRAPHS

FROM: 03/01/85 TO: 24/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

20-50% F.S.

2-4% 4-6% > 6%

0-2%

0-2% 2-4% 4-6% > 6%

50-100% F.S.

0

0-2% 2-4% 4-6% > 6%

0-20% F.S.

PH QUALITY CONTROL DATA FROM 24/01/85 TO 19/12/85

Lab: Dorset

Analytical Range: 0.00 to 14.00

CALIBRATION (	CONTROL
---------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	116	6.86	6.87	0.01	0.012
b		116	4.00	4.01	0.01	0.012
a+b		116	10.86	10.88	0.02	0.019
a-b		116	2.86	2.87	0.01	0.015

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.012 S/Sw: 1.13

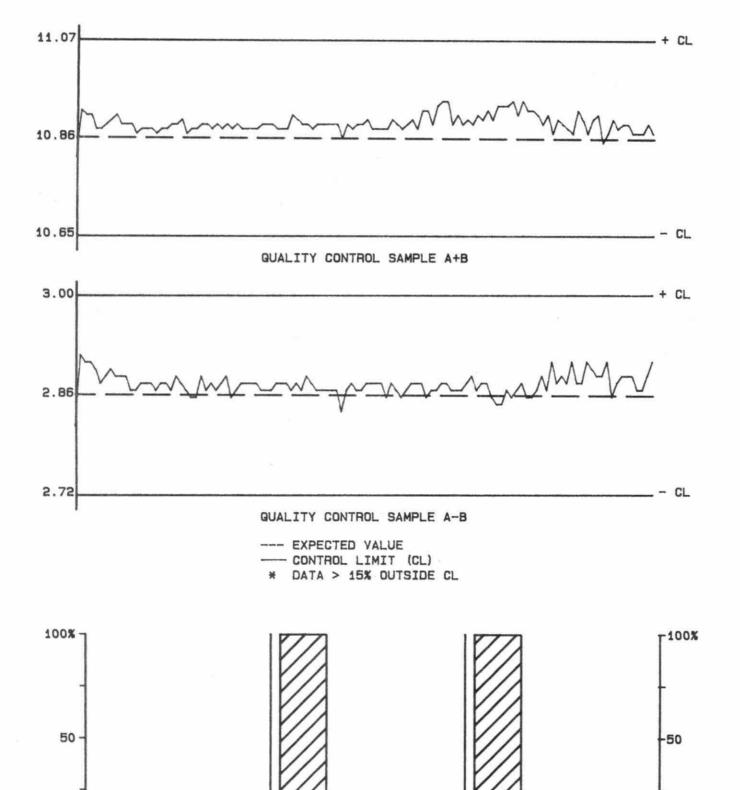
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 to 11.07 for A+B 2.72 to 3.00 for A-B

DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	0	0.00 - 4.00	N/A	N/A
	40	4.00 - 5.50	0.014	0.2
	194	5.50 - 7.00	0.025	0.4
	6	7.00 - 8.50	0.033	0.4
)4	Ø	8.50 - 14.00	N/A	N/A
	240	Overal1	0.024	N/A

## QUALITY CONTROL GRAPHS

FROM: 24/01/85 TO: 19/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

20-50% F.S.

2-4% 4-6% > 6%

0-2% 2-4% 4-6% > 6%

50-100% F.S.

0-2%

0

2-4% 4-6% > 6%

0-20% F.S.

#### *** PH ***

#### IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/05/79

LIS Test Name Code: PH Units : Dimensionless

Work Station Code : PHACD Unit Code : Nil

Method Code : 002AII Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

N.B. Gran and total fixed endpoint acidity are determined simultaneously.

#### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, eg, QCA

#### MODIFICATIONS:

01/04/82- Sample volume was decreased from 100.0 to 10.0 mL. 01/05/83- System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

PH QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 0.00 to 14.00

COLI	DDAT	TONE	CONTROL	-
one i	DKUI	TOIN	CUNIKUL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a.	2	148	6.86	6.87	0.01	0.008
ь	2	148	4.01	4.01	-0.00	0.005
a+b	2	148	10.87	10.88	0.01	0.012
a-b		148	2.85	2.86	0.01	0.007

s.d.(AB): Sw(within run): 0.005 S(between runs): 0.007 S/Sw: 1.35

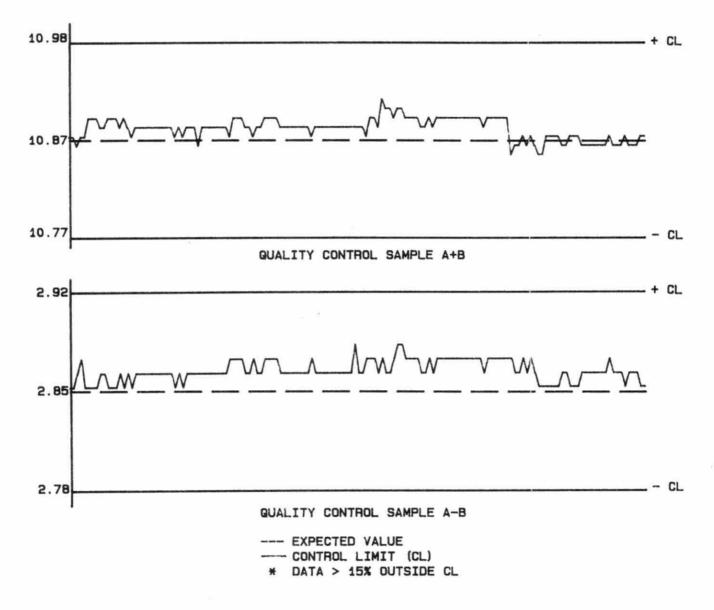
On any given day the calibration is accepted if the values obtained lie within the ranges:

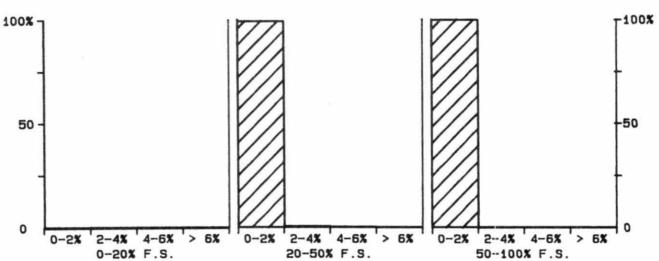
10.76 to 10.97 for A+B 2.78 to 2.92 for A-B

DUPLICATES:	Number of	Samp 1	e	Mean(2)	Coefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	0	0.00 -	3.00	N/A	N/A
	37	3.00 -	4.00	0.025	0.6
	244	4.00 -	5.00	0.027	0.6
	88	5.00 -	7.00	0.089	1.5
	8	7.00 -	14.00	0.085	1.1
	377	Overa	11	0.051	NZA

## QUALITY CONTROL GRAPHS

FROM: 02/01/85 TO: 23/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### *** PH ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 09/07/80

LIS Test Name Code: PH Units : Dimensionless

Work Station Code : RATS Unit Code : Nil

Method Code : 003AI2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic, filled

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

N.B. Alkalinity (Gran and total fixed endpoint) are determined simultaneously

#### INSTRUMENTATION:

RATS: Automated titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, sulfuric acid and sodium carbonate solutions, eg, QCA

Drift : In run standard, 20% tap water - throughout run. Diluted tap water (20 % V/V)

#### MODIFICATIONS:

02/03/84- QC program at this workstation was expanded to include pH and total fixed endpoint alkalinity. Preparation and storage of QC solutions was modified. As shown by the following QCA-B data, the attempt to check pH calibrations by monitoring the pH values of the dilute alkalinity standards failed. Buffers will be utilized in '85.

16/03/84- Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped linners was recommended for sampling.

09/05/85- RATS- River Automated Titration System. Designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer (DCI) capabilities.

No data summary is available for period not covered in report.

#### PH QUALITY CONTROL DATA FROM 09/05/85 TO 31/12/85

Lab: Rivers and Lakes

Analytical Range: 0.00 to 14.00

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	97	4.45	4.47	0.02	0.018
b	1	97	3.73	3.72	-0.01	0.027
a+b		96	8.18	8.20	0.02	0.035
a-b	1	96	0.72	0.75	0.03	0.029

s.d.(AB): Sw(within run): 0.021 S(between runs): 0.023 S/Sw: 1.12

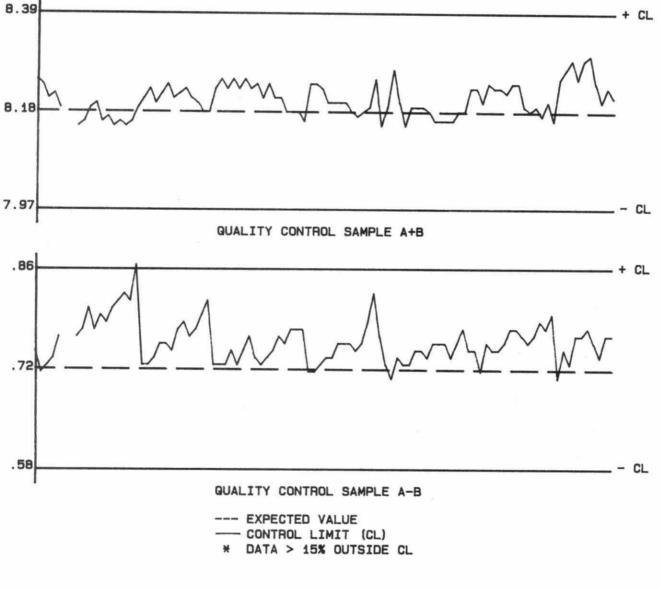
On any given day the calibration is accepted if the values obtained lie within the ranges:

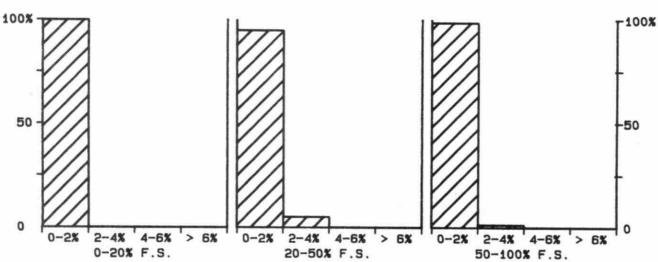
7.97 to 8.39 for A+B 0.58 to 0.86 for A-B

DUPLICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Spa	ın	s.d.	of var.(%)
	9	0.00 -	5.00	0.038	0.9
	13	5.00 -	6.00	0.111	2.0
	38	6.00 -	7.00	0.116	1.7
	211	7.00 -	9.00	0.066	0.8
	9	9.00 - 1	4.00	0.108	1.0
	280	Overal 1	Ľ.	0.078	N/A

# QUALITY CONTROL GRAPHS

FROM: 09/05/85 TO: 31/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### *** PH ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '70

LIS Test Name Code: PH Units " Dimensionless

Work Station Code : SBPH Unit Code : Nil

Method Code : 001AI1 Supervisor : P. Campbell

Sample Type/Matrix: Sewage, Industrial Waste, Effluents

#### SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (50 mL) at room temperature. Stirring rate and room temperature range are uniform for all samples and standards.

#### INSTRUMENTATION:

pH meter, stirrer, glass electrode

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

#### CONTROLS:

Calibration: 2 standard buffers

QUALITY CONTROL DATA FROM 07/01/85 TO 16/12/85

Lab: Sewage and Industrial Waste

Analytical Range: 0.00 to 14.00

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		121	9.00	8.98	-0.02	0.037
ь	:	121	4.00	4.00	0.00	0.022
a+b	1	121	13.00	12.98	-0.02	0.045
a-b		121	5.00	4.98	-0.02	0.041

s.d.(AB): Su(within run): 0.029 S(between runs): 0.030 S/Su: 1.05

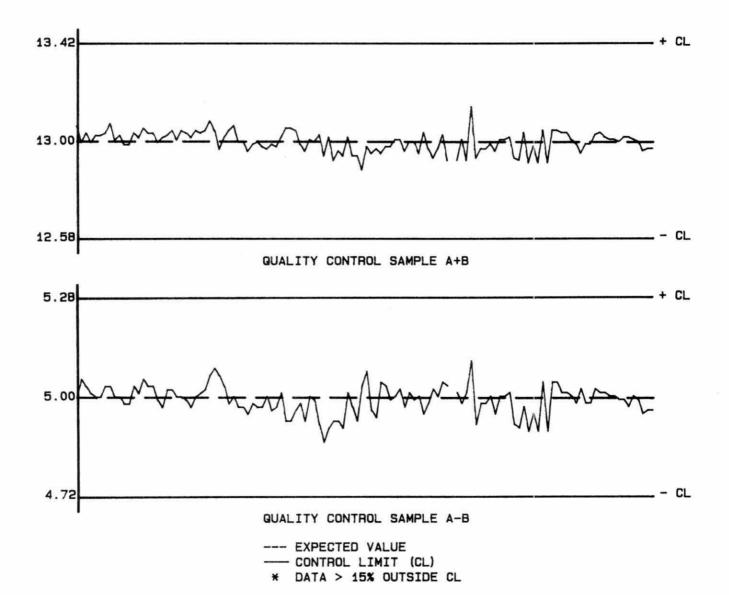
On any given day the calibration is accepted if the values obtained lie within the ranges:

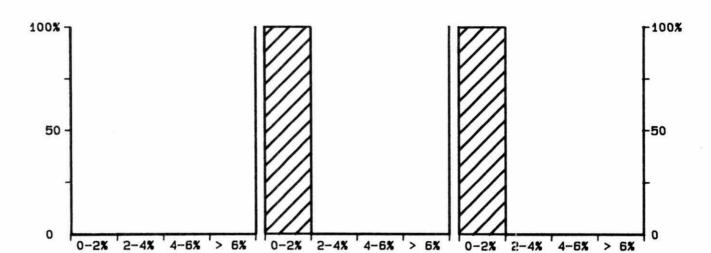
12.58 to 13.42 for A+B 4.72 to 5.28 for A-B

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	9	0.00 - 5.00	0.054	1.3
	45	5.00 - 7.00	0.034	0.5
	99	7.00 - 8.00	0.026	0.3
	28	8.00 - 9.00	0.029	0.3
	2	9.00 - 14.00	0.050	0.4
	183	Overal1	0.031	N/A

## QUALITY CONTROL GRAPHS

FROM: 07/01/85 TO: 16/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

20-50% F.S.

50-100% F.S.

0-20% F.S.

#### *** PHENOLICS - REACTIVE ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74

LIS Test Name Code: PHNOL Units : ug/L as Phenol

Work Station Code : ROPHEN Unit Code : 063704

Method Code : 002BC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required: 250 mL Container : Glass

Preservative : Copper sulphate-phosphoric acid

Other : Special bottle (with white cap) containing preservative is

available

#### ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide. Approximate absorbance: 0.03 at the 50 ug/L as phenol level.

#### INSTRUMENTION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.2

Detection Criterion (T): 1.0

#### CALIBRATION:

BL plus 1 standard

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard

#### NOTES:

A report identifying reactive phenolics is available on request.

#### PHENOLICS - REACTIVE QUALITY CONTROL DATA FROM 04/01/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 1.0 to 50.0 ug/L as PHENOL

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		95	40.0	40.0	0.0	0.34
b	2	95	10.0	10.3	0.3	0.31
a+b		95	50.0	50.3	0.3	0.42
a-b		95	30.0	29.8	-0.2	0.50

s.d.(AB): Sw(within run): 0.35 S(between runs): 0.33 S/Sw: 0.92

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 47.8 to 52.2 for A+B 28.5 to 31.5 for A-B

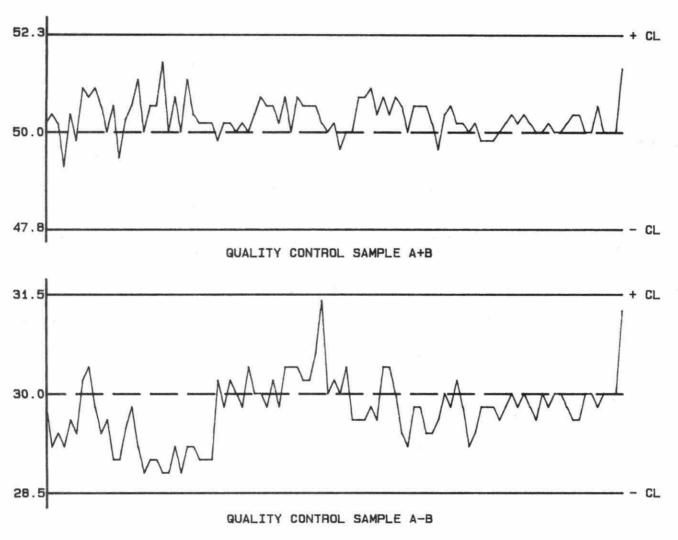
DUPLICATES	Number of Data Pairs	Sample Concn Spa	n	Mean(2)	Coefficient of var.(%)
	188	0.0 -	5.0	0.35	39.9
	13	5.0 -	10.0	0.45	5.7
	15	10.0 -	25.0	1.14	6.9
	11	25.0 -	50.0	0.95	2.8
	227	Overall	l	0.49	N/A

#### DETECTION CRITERION: 1.0

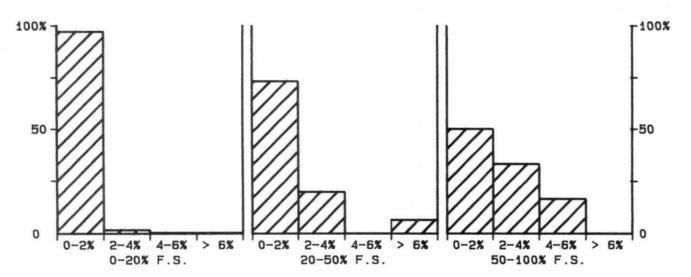
OTHER CHECKS:	Number of Data		Data Mean	Standard(1) Deviation
Std Cal		92	918	9.2
Long Term Blank	:	95	0.2	0.11

# QUALITY CONTROL GRAPHS PHENOLICS - REACTIVE (UG/L AS PHENOL)

FROM: 04/01/85 TO: 30/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 UG/L AS PHENOL

### *** PHOSPHORUS - REACTIVE ORTHOPHOSPHATE ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/79
LIS Test Code Name: PPO4FR Units : mg/L as P
Work Station Code : RNDNP Unit Code : 064815
Method Code : 103DC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Orthophosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.2 at the 0.13 mg/L as P level.

N.B. Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.0005

Detection Criterion (T): 0.0030

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : BL plus 1 standard

### MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G). Reduction period was reduced from 4 to 2 min. to lessen danger of poly phosphate conversion to orthophosphate during analysis.

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

### PHOSPHORUS - REACTIVE ORTHOPHOSPHATE QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

Lab: Rivers and Lakes

Analytical Range: 0.0028 to 0.1250 mg/L as P

TO!	TODAT	LADIT	COAL	TROL:
	IDKUI	TOIA	CUN	INUL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	139	0.1000	0.1005	0.0005	0.00219
b		139	0.0250	0.0259	0.0009	0.00157
a+b	2	139	0.1250	0.1263	0.0013	0.00275
a-b		139	0.0750	0.0746	-0.0004	0.00263
C	:	139	0.0250	0.0259	0.0009	0.00156
d		139	0.0125	0.0127	0.0002	0.00089
c+d	:	139	0.0375	0.0385	0.0010	0.00234
c-d	:	139	0.0125	0.0132	0.0007	0.00100

s.d.(AB): Sw(within run): 0.00186 S(between runs): 0.00191 S/Sw: 1.02 s.d.(CD): Sw(within run): 0.00071 S(between runs): 0.00127 S/Sw: 1.80

On any given day the calibration is accepted if the values obtained lie within the ranges:

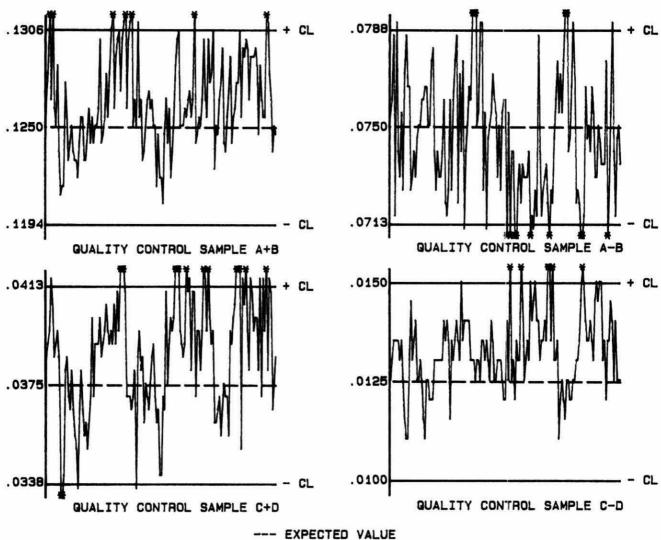
0.1194 to 0.1306 for A+B 0.0712 to 0.0787 for A-B 0.0337 to 0.0412 for C+D 0.0100 to 0.0150 for C-D

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	234	0.0000 - 0.0100	0.0009	28.1
	39	0.0100 - 0.0200	0.0012	8.8
	51	0.0200 - 0.0500	0.0097	31.5
	51	0.0500 - 0.1250	0.0136	17.7
	375	Overall	0.0062	N/A

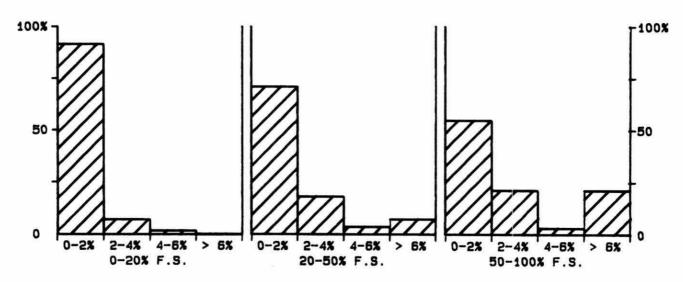
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Me an	Deviation
Long Term Blank	: 139	0.0010	0.00049

# QUALITY CONTROL GRAPHS PHOSPHORUS - REACTIVE ORTHOPHOSPHATE (MG/L AS P)

FROM: 03/01/85 TO: 28/01/86



--- EXPECTED VALUE
---- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .125 MG/L AS P

### *** PHOSPHORUS - REACTIVE ORTHOPHOSPHATE ***

### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/79
LIS Test Name Code: PPO4FR Units : mg/L as P
Work Station Code: SNH3P Unit Code : 064815
Method Code : 103BC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent. Approximate absorbance: 0.4 at the 4.0 mg/L as P level.

N.B. Ammonia plus ammonium is determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.05

### CALIBRATION:

BL plus 2 standards

### CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL plus 2 standards

## PHOSPHORUS - REACTIVE ORTHOPHOSPHATE QUALITY CONTROL DATA FROM 03/01/85 TO 30/12/85

Lab: Sewage and Industrial Waste Analytical Range: 0.05 to 10.00 mg/L as P

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	136	7.00	6.93	-0.07	0.104
ь	1	136	3.50	3.45	-0.05	0.059
a+b	t	136	10.50	10.37	-0.13	0.141
a-b	1	136	3.50	3.48	-0.02	0.093
c	1	137	3.50	3.54	0.04	0.061
d	2	136	0.70	0.70	0.00	0.031
c+d	1	136	4.20	4.24	0.04	0.073
c-d		136	2.80	2.83	0.03	0.063

s.d.(AB): Sw(within run): 0.066 S(between runs): 0.085 S/Sw: 1.29 s.d.(CD): Sw(within run): 0.045 S(between runs): 0.048 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

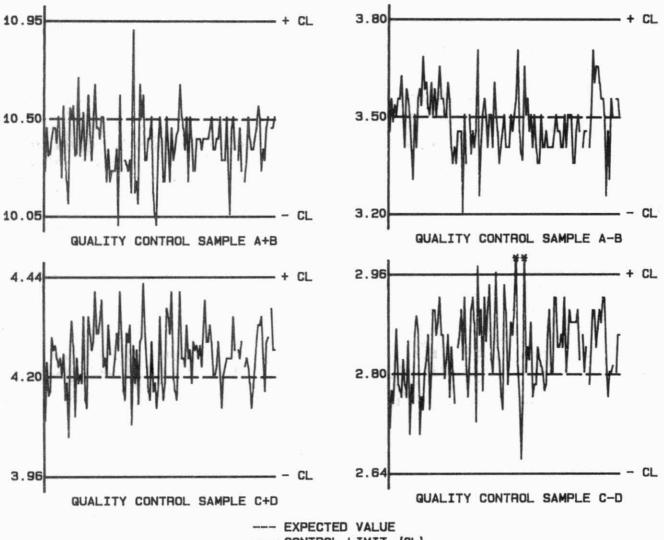
10.05 to 10.95 for A+B 3.20 to 3.80 for A-B 3.96 to 4.44 for C+D 2.64 to 2.96 for C-D

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	136	0.00 - 0.40	0.017	36.3
	20	0.40 - 1.00	0.042	6.0
	22	1.00 - 2.00	0.083	5.4
	24	2.00 - 4.00	0.068	2.4
	17	4.00 - 10.00	0.132	2.3
	219	Overal1	0.054	N/A

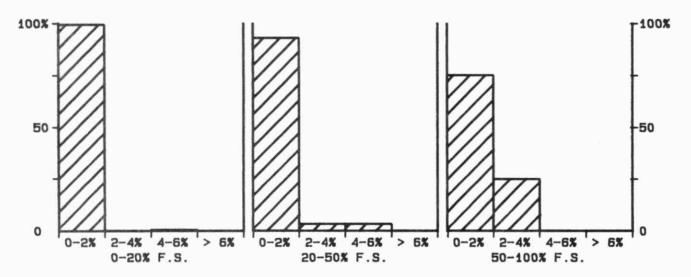
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal	:	82	665	44.4
Long Term Blank		113	0.01	0.000

# QUALITY CONTROL GRAPHS PHOSPHORUS - REACTIVE ORTHOPHOSPHATE (MG/L AS P)

FROM: 03/01/85 TO: 30/12/85



--- EXPECTED VALUE
---- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 10 MG/L AS P

### *** PHOSPHORUS - TOTAL ***

Laboratory

: Dorset

Method Introduced: 22/03/79

Supervisor

: F. Tomassini

Units

ug/L as P

Sample Type/Matrix: Streams, Lakes, Precipitation

#### SAMPLING:

Quantity Required: 35 mL

Container

: Specially marked Pyrex culture tubes with Teflon-lined caps

### ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121 C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.3 at the 200 ug/L as P level.

### INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 0.8

### CALIBRATION:

BL plus 2 undigested standards

### CONTROLS:

Calibration: LTBL plus 4 undigested standards, eg, QCA

Recovery : 3 digested BL plus 4 digested standards, eg, R1

Drift : BL every 10 samples and BL plus 2 undigested standards every 20

samples.

### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

## PHOSPHORUS - TOTAL QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

Lab: Dorset

Analytical Range: 0.8 to 200 ug/L as P

CALIBRA	HION	CONTROL: Number of Data		xpected Concn	Av.Con Measur		Av.Bias		andard(1)
			-						
a	:	164		171	171		Ø		1.6
b	I	164		57	57		Ø		0.6
a+b	t	164	9	228	229		1		2.0
a-b	1	164		114	114		Ø		1.2
C	:	164		17.1	17.1		0.0		0.20
d	E	164		5.7	5.7		0.0		0.09
c+d	:	164		22.8	22.9		0.1		0.26
c-d	:	164		11.4	11.4		0.0		0.17
s.d.(AE	3): 5	Sw(within	run):	0.8	S(between	runs):	1.2	S/Sw I	1.42
s.d.(CD	)): (	Sw(within	run):	0.12	S(between	runs):	0.16	S/SHI	1.29

On any given day the calibration is accepted if the values obtained lie within the ranges:

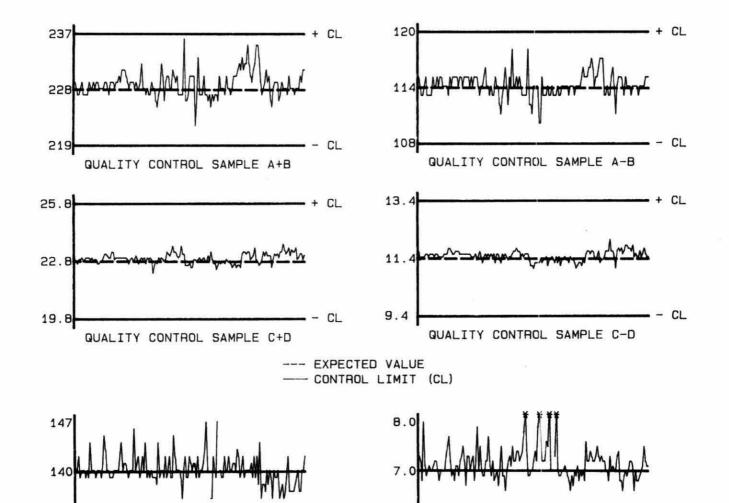
219 to 237 for A+B 108 to 120 for A-B 19.8 to 25.8 for C+D 9.4 to 13.4 for C-D

RECOVERIES		Number of Data		Cor	pected ncn	Av.Conc. Measured	Standard(1) Deviation
r i	:	162			140	140	2.0
					37.3 LT (450)	140	2.0
r2	2	163			70	70	1.3
r3	:	164			14.0	14.2	0.46
r4	1	162			7.0	7.2	0.47
DUPLICATES:	Numbe	r of	S	a.mp	l e	Mean(2)	Coefficient
	Data	Pairs	Con	en s	Span	s.d.	of var.(%)
	83		0.0	-	5.0	0.28	8.1
	156		5.0	-	10.0	0.65	8.9
	128		10.0	-	20.0	0.66	4.5
	76		20	-	50	2.4	7.7
	20		50	-	200	2.1	2.5
	463		0	ver	a.1 1	1.2	N/A

OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Std. Cal.	:	153	524	19.7
Long Term Bla	nK I	164	0.0	0.14
Digested Bland	Κ :	164	0.9	0.24

# QUALITY CONTROL GRAPHS PHOSPHORUS - TOTAL (UG/L AS P )

FROM: 03/01/85 TO: 20/12/85



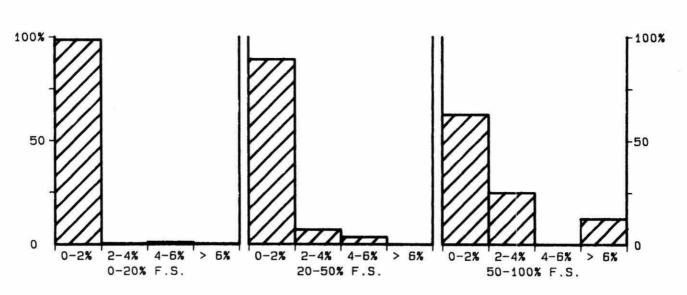
* DATA > 15% DUTSIDE CL

6.0

RECOVERY SAMPLE R4

133

RECOVERY SAMPLE R1



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 UG/L AS P

### *** PHOSPHORUS-TOTAL ***

### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/79
LIS Test Name Code: PPUT Units : mg/L as P
Work Station Code : RTNP Unit Code : 064815
Method Code : 504AC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the 0.20 mg/L as P level. N.B. Total Kjeldahl nitrogen is determined simultaneously.

### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

-Data capture, reduction, and processing via a multi-stage microcomputer system REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.001

Detection Criterion (T): 0.007

### CALIBRATION:

BL plus 3 undigested standards

### CONTROLS:

Calibration: LTBL plus 2 undigested standards, eg, QCA

Recovery : 3 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL plus 1 undigested standard

### MODIFICATIONS:

15/08/83 - Microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic.

### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

## PHOSPHORUS-TOTAL QUALITY CONTROL DATA FROM 01/01/85 TO 23/12/85

Lab! Rivers and Lakes

Analytical Range: 0.007 to 0.200 mg/L as P

### CALIBRATION CONTROL:

	Number		Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	158	0.150	0.149	-0.001	0.0017
b	•	158	0.050	0.050	0.000	0.0020
a+b		158	0.200	Ø.199	-0.001	0.0030
a-b		158	0.100	0.699	-0.001	0.0021

s.d.(AB): Sw(within run): 0.0015 S(between runs): 0.0019 S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.191 to 0.209 for A+B 0.094 to 0.106 for A-B

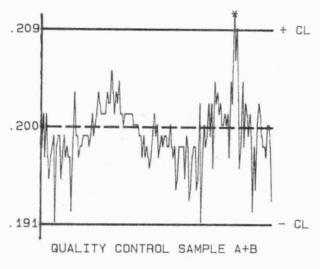
RECOVERIES	9	Number of Data		Av.Conc. Measured	Standard(1) Deviation
r1	1	158	0.140	0.137	0.0047
r2	•	158	0.084	0.082	0.0028
r3		158	0.028	0.028	0.0023
DUPL ICATES:	Number	of	Sample	Mean(2)	Coefficient
	Data P	airs	Concn Span	s.d.	of var.(%)
	100				

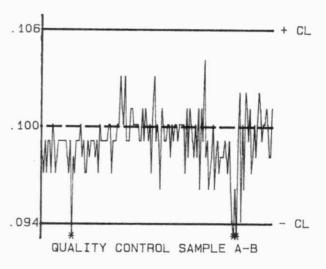
196	0.000 -	0.020	0.0023	24.5
101	0.020 -	0.050	0.0039	11.7
38	0.050 -	0.100	0.0068	10.1
25	0.100 -	0.200	0.0101	7.5
360	Over	11	0.0044	N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	2	153	0.001	0.0003
Digested Blank	:	156	0.002	0.0021

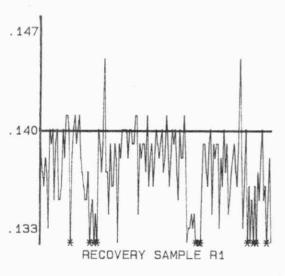
# QUALITY CONTROL GRAPHS PHOSPHORUS-TOTAL (MG/L AS P)

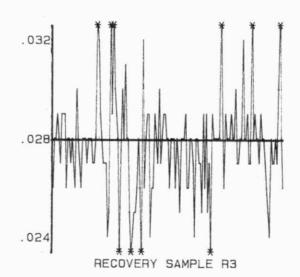
FROM: 01/01/85 TO: 23/12/85



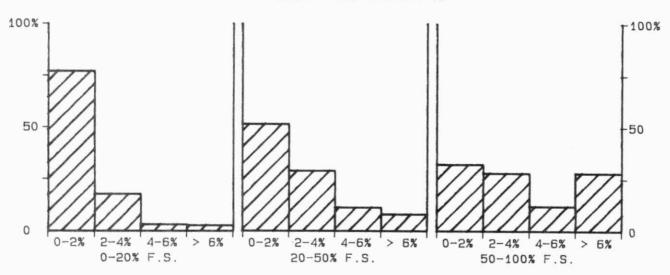


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): .2 MG/L AS P

### *** PHOSPHORUS - TOTAL ***

### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/79
LIS Test Name Code: PPUT Units : mg/L as P
Work Station Code: STKNP Unit Code : 084815
Method Code : 5048C2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Effluents,
Leachates

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.4 at the 2.0 mg/L as P level. N.B. Total Kjeldahl nitrogen is determined simultaneously.

### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W) : 0.01

Detection Criterion (T): 0.03**

### CALIBRATION:

BL plus 2 undigested standards

### CONTROLS:

Calibration : LTBL plus 4 undigested standards, eg, QCA

Recovery | 2 digested BL plus 3 digested standards in duplicate, eg, R1

Drift | BL plus 2 undigested standards

### MODIFICATIONS:

01/10/85 -High range added, full scale changed from 2 to 5 mg/L as P. New calibration controls added. Calibration control results collected before high range was implemented are included in plot.

### NOTES:

System is calibrated with undigested standards.

**Minimum dilution is 50% (i.e. factor of two). Therefore, minimum increment and detection criterion are actually 0.02 and 0.06 respectively.

## PHOSPHORUS - TOTAL QUALITY CONTROL DATA FROM 03/01/85 TO 31/12/85

Lab! Sewage and Industrial Waste Analytical Range: 0.03 to 5.00 mg/L as P

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	115	3.50	3.47	-0.03	0.060
b	E	114	1.40	1.36	-0.04	0.039
a+b		114	4.90	4.83	-0.07	0.089
a-b	1	114	2.10	2.10	0.00	0.048
c		164	1.400	1.418	0.018	0.0248
d	2	163	0.280	0.286	0.006	0.0198
c+d	:	163	1.680	1.704	0.024	0.0393
c-d	E .	163	1.120	1.132	0.012	0.0221

s.d.(AB): Sw(within run): 0.034 S(between runs): 0.051 S/Sw: 1.49 s.d.(CD): Sw(within run): 0.0156 S(between runs): 0.0225 S/Sw: 1.44

On any given day the calibration is accepted if the values obtained lie within the ranges:

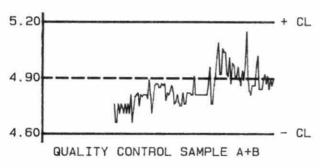
4.60 to 5.20 for A+B 1.90 to 2.30 for A-B 1.560 to 1.800 for C+D 1.040 to 1.200 for C-D

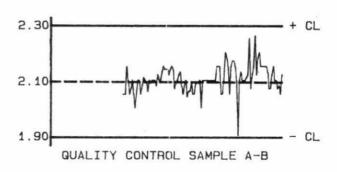
RECOVERIES		Number of Data		- 22 0	pected ncn	Av.Conc. Measured	Standard(1) Deviation
r1	1	106			3.50	3.40	0.103
r2	f	147			1.40	1.39	0.036
r3		147			0.70	0.67	0.035
DUPLICATES:	Number Data 1			a.mp	le Span	Mean(2)	Coefficient
						3.4.	of var.(%)
	190		0.000	-	0.20	0.009	19.7
	54		0.200	-	0.40	0.039	14.4
	63		0.40	-	1.00	0.025	3.8
	30		1.00	-	2.00	0.064	4.6
	7		2.00	-	5.00	0.077	3.2
	344		0	ver	a.1 1	0.029	N/A

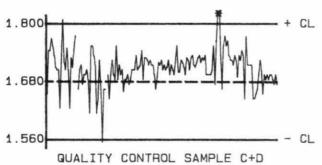
OTHER CHECKS!		Number	Data	Standard(1)
		of Data	Me an	Deviation
Std. Cal	:	141	456	28.7
Long Term Blank		147	0.007	0.0057
Digested Blank	:	129	0.007	0.0035

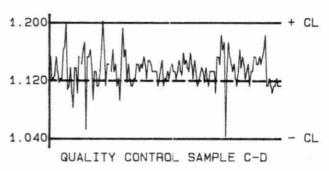
### QUALITY CONTROL GRAPHS PHOSPHORUS - TOTAL (MG/L AS P)

FROM: 03/01/85 TO: 31/12/85

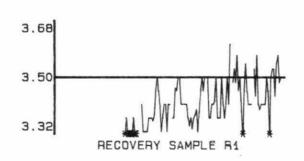


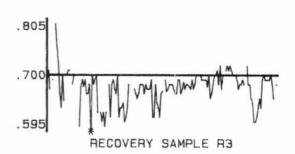




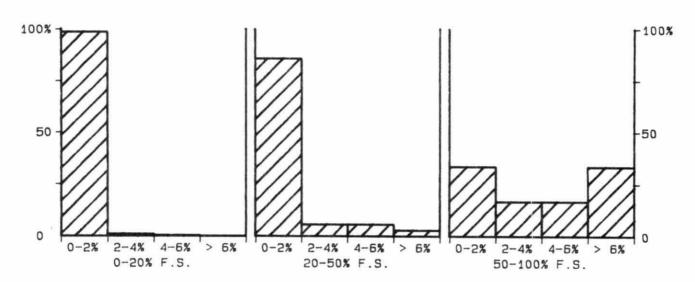


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS P

### *** POTASSIUM ***

### IDENTIFICATION:

Laboratory : Domestic Water

Method Introduced: Before '74

LIS Test Name Code: KKUR

Units : mg/L as K

Work Station Code : WNAK

Unit Code : 064819

Method Code : 002BA1

Supervisor

:M. Rawlings

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

### SAMPLING:

Quantity Required: 50 mL

odancity Reduired: 30 MF

Container

: Glass or plastic

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: 0.15 at 10 mg/L as K level.

### INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical anges are obtained from the output of the AAS.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T):0.15

### CALIBRATION:

BL plus 2 standards.

### CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

## POTASSIUM QUALITY CONTROL DATA FROM 02/01/85 TO 31/12/85

Lab: Domestic Water

Analytical Range: 0.15 to 40.0 mg/L as K

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1)
				reasured		Deviation
a.	:	100	26.0	26.2	0.2	0.31
ь	1	100	6.5	6.5	0.0	0.14
a+b	1	100	32.5	32.6	0.1	0.33
a-b	1	100	19.5	19.7	0.2	0.36
c		100	6.50	6.45	-0.05	0.107
ď	:	100	1.30	1.33	0.03	0.058
c+d	1	100	7.80	7.79	-0.01	0.141
c-d	:	100	5.20	5.12	-0.08	0.099

s.d.(AB): Sw(within run): 0.25 S(between runs): 0.24 S/Sw: 0.94 s.d.(CD): Sw(within run): 0.070 S(between runs): 0.086 S/Sw: 1.23

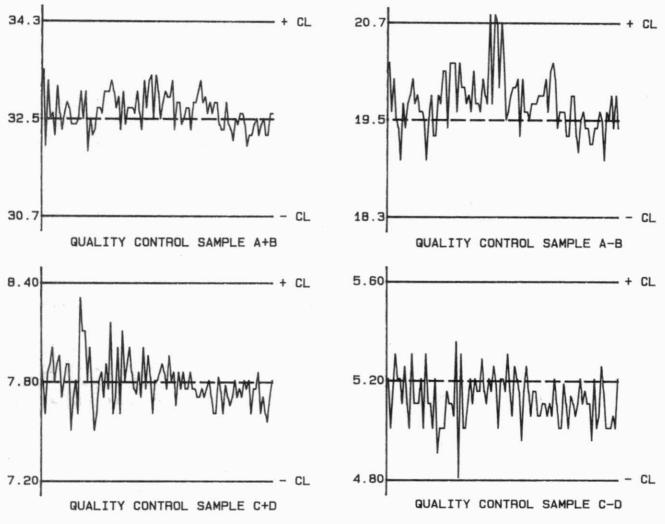
On any given day the calibration is accepted if the values obtained lie within the ranges:

30.7 to 34.3 for A+B 18.3 to 20.7 for A-B 7.20 to 8.40 for C+D 4.80 to 5.60 for C-D

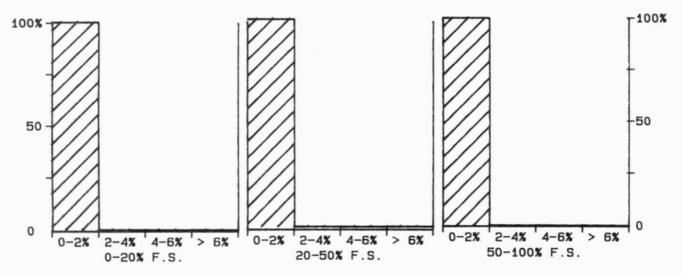
DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	61	0.00 - 1.00	0.051	7.4
	116	1.00 - 2.00	0.065	4.4
	51	2.00 - 5.00	0.094	2.9
	16	5.0 - 10.0	0.11	1.6
	10	10.0 - 40.0	0.22	1.4
	254	Overal1	0.08	N/A

### QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 02/01/85 TO: 31/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 MG/L AS K

### POTASSIUM ***

### IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 18/05/79

LIS Test Name Code: KKUR

: mg/L as K Units

Work Station Code : PRAA

Unit Code # 064819

: 002EA1 Method Code

Supervisor

: M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

#### SAMPLING:

Quantity Required: 5 mL

: Polystyrene

### ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 766.5 nm with an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: 0.5 at the 1.00 mg/L level.

### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer(AAS) system

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.005

Detection Criterion (T): 0.025

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples, 2 standards every 20 samples.

## POTASSIUM QUALITY CONTROL DATA FROM 08/01/85 TO 20/12/85

Lab: Precipitation

Analytical Range: 0.025 to 1.00 mg/L as K

CAL.	TRRAT	MOT	CON	TROL:
-	ADMI	LOIA	COLA	I PULL .

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	71	0.600	0.595	-0.005	0.0106
ь	:	72	0.100	0.096	-0.004	0.0101
a+b	1	69	0.700	0.691	-0.009	0.0139
ab		69	0.500	0.499	-0.001	0.0148

s.d.(AB): Sw(within run): 0.0105 S(between runs): 0.0104 S/Sw: 0.99

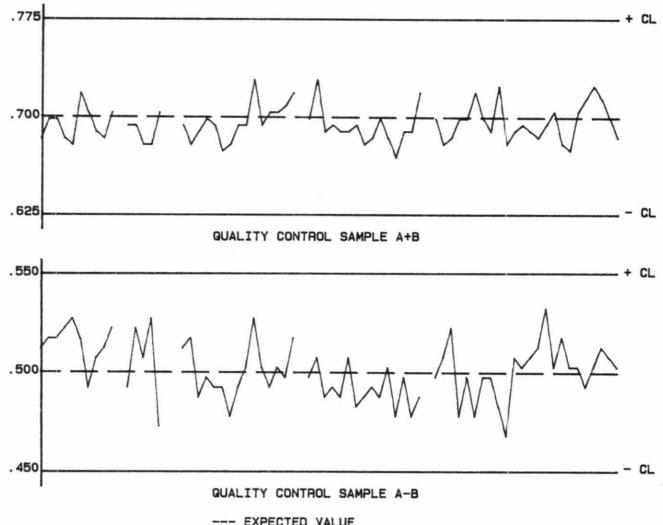
On any given day the calibration is accepted if the values obtained lie within the ranges:

0.625 to 0.775 for A+B 0.450 to 0.550 for A-B

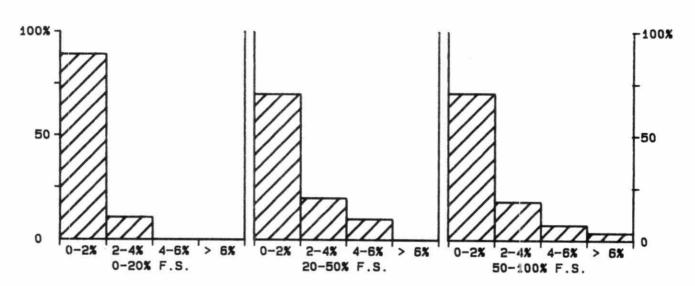
DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	114	0.000 - 0.100	0.0082	22.2
	16	0.100 - 0.200	0.0110	8.0
	32	0.20 - 1.00	0.012	2.1
	162	Overal1	0.009	N/A

### QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 08/01/85 TO: 20/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 1 MG/L AS K

### *** POTASSIUM ***

### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: KKUR Units : mg/L as K

Work Station Code : RMAAS Unit Code : 064819

Method Code : 002CA1,002DA1 Supervisor : J. Crowther Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.
Approximate absorbance: RMAAS: 0.8 full scale value

#### INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

#### REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.01 Detection Criterion (T):0.12,0.12,0.05

### CALIBRATION:

BL plus 10 standards

### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA for each analytical range

Drift : BL plus 1 standard for each analytical range

### MODIFICATIONS:

01/12/81- Calibration range became 5.00 mg/L full scale; second analytical range was dropped.

01/03/84- Analytical range(RMNAKL) was added; full scale:1.00 mg/L. This range is currently restricted to special programs.

01/09/84- Analytical range(RMNAKH) was increased from 5.00 to 10.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Sodium is no longer determined simultaneously.

25/09/85- Calibration range stayed at 10.0 mg/L but second analytical range was dropped. Concentration of QC solutions was modified. Microcomputer controlled system.

### NOTES:

Three analytical ranges were used during 1985: 1.00, 10.0, and 10.0 mg/L full scale (difference in latter two ranges was choice of control solution concentrations) with the respective detection criteria shown above.

## POTASSIUM QUALITY CONTROL DATA FROM 03/01/85 TO 09/09/85

Lab: Rivers and Lakes

Analytical Range: 0.12 to 10.00 mg/L as K

CAL	PPAT	MOLT	CON	TROL:
Unt.	IDRH	TOLA	CUIN	I RUL .

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	35	7.50	7.54	0.04	0.170
b	:	35	2.50	2.52	0.02	0.062
a+b		35	10.00	10.06	0.06	0.196
a-b	:	35	5.00	5.01	0.01	0.164

s.d.(AB): Sw(within run): 0.116 S(between runs): 0.128 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

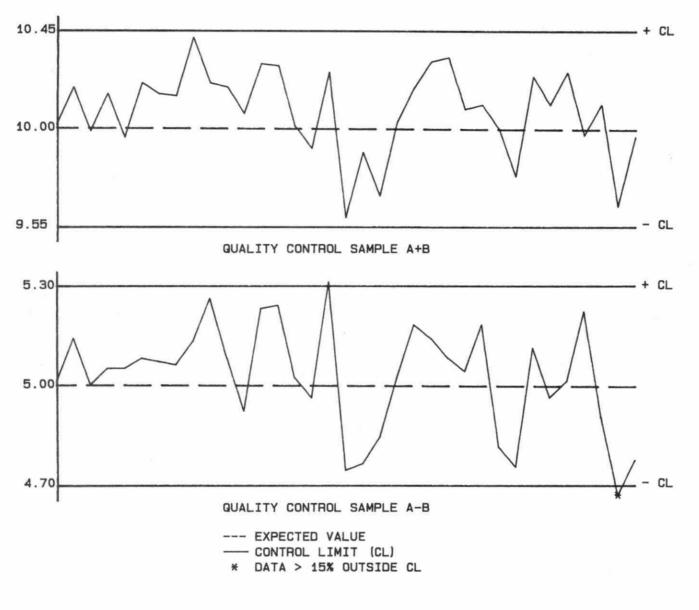
9.55 to 10.45 for A+B 4.70 to 5.30 for A-B

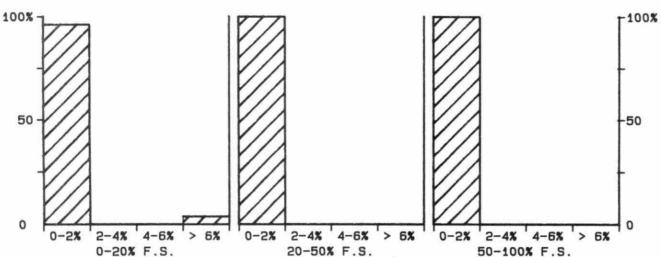
DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	41	0.00 - 0.50	0.039	19.7
	16	0.50 - 1.00	0.030	4.2
	21	1.00 - 2.50	0.044	3.0
	15	2.50 - 5.00	0.063	1.8
	2	5.00 - 10.00	0.081	1.5
	95	Overal1	0.045	N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance		28	0.94	0.141
Long Term Blank	:	0	N/A	N/A

# QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 03/01/85 TO: 09/09/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 10 MG/L AS K

## POTASSIUM QUALITY CONTROL DATA FROM 25/09/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.05 to 10.00 mg/L as K

COL	TODAT	LADI	COAR	ROL:
LITE.	IBRHI	IUN	CUNI	KUL .

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	29	8.00	8.06	0.06	0.152
ь		29	0.70	0.70	0.00	0.019
a+b	:	29	8.70	8.77	0.07	0.155
a-b	1	29	7.30	7.36	0.06	0.152

s.d.(AB): Sw(within run): 0.107 S(between runs): 0.108 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

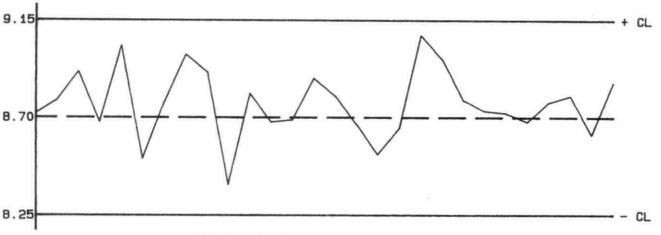
8.25 to 9.15 for A+B 7.00 to 7.60 for A-B

DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	46	0.00 - 0.50	0.017	5.7
	23	0.50 - 1.00	0.037	5.4
	8	1.00 - 2.00	0.014	0.9
	10	2.00 - 5.00	0.077	2.2
	1	5.00 - 10.00	N/A	N/A
	88	Overall	0.038	N/A

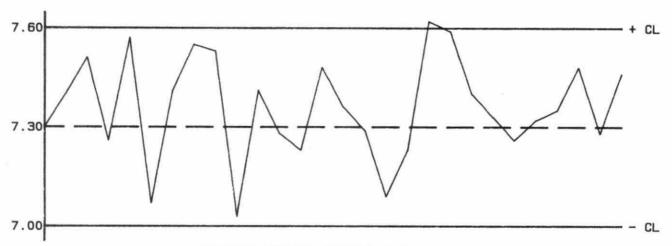
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	Ø	N/A	N/A
Long Term Blank	:	Ø	N/A	N/A

### QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 25/09/85 TO: 30/12/85

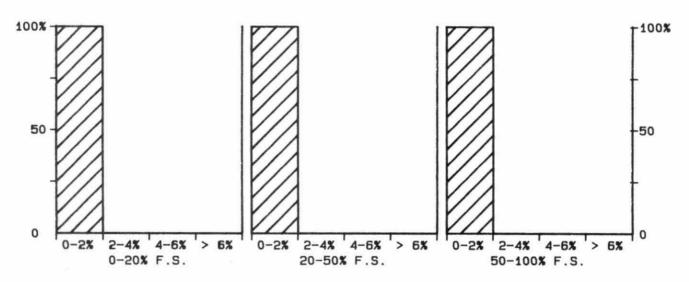


QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B

--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS K

## POTASSIUM QUALITY CONTROL DATA FROM 07/01/85 TO 10/09/85

Lab: Rivers and Lakes

Analytical Range: 0.12 to 1.00 mg/L as K

COL	PERTICAL	CONTROL
LITTL	IDKHIIUN	LUNINUL

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	42	0.75	0.74	-0.01	0.016
ь		42	0.25	0.25	-0.00	0.013
a+b		42	1.00	0.99	-0.01	0.023
a-b	1	42	0.50	0.50	-0.00	0.018

s.d.(AB): Sw(within run): 0.013 S(between runs): 0.015 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

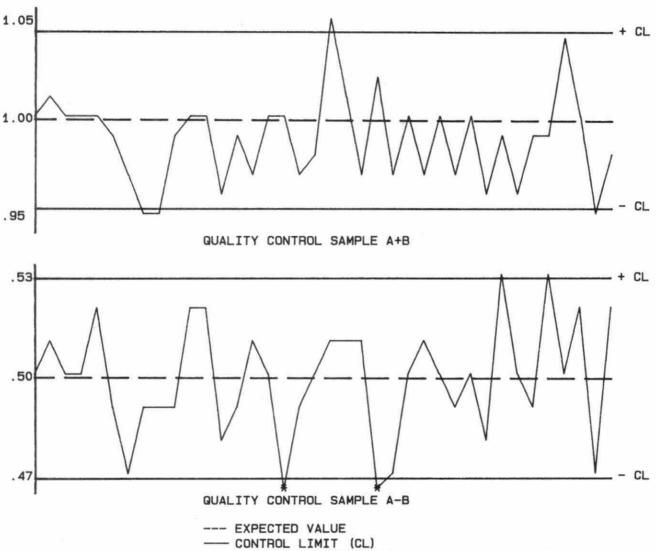
0.95 to 1.05 for A+B 0.47 to 0.53 for A-B

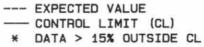
DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	4	0.00 - 0.10	0.039	78.1
	16	0.10 - 0.25	0.023	12.8
	63	0.25 - 0.50	0.028	7.3
	31	0.50 - 1.00	0.043	6.7
	114	Overall	0.033	N/A

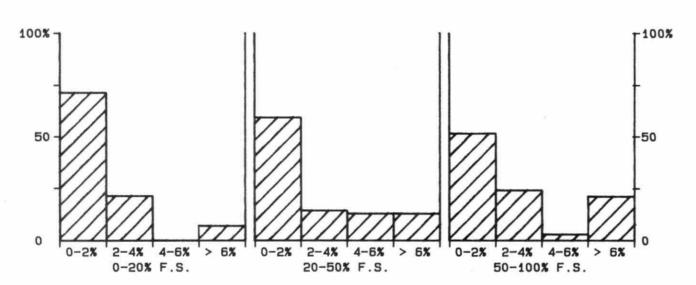
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	30	0.48	0.230
Long Term Blank		Ø	N/A	N/A

### QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 07/01/85 TO: 10/09/85







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1 MG/L AS K

### *** SILICON - REACTIVE SILICATES ***

### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/02/75
LIS Test Name Code: SIO3UR Units : mg/L as Si
Work Station Code: RMSICL Unit Code : 064814
Method Code : 001BC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates

#### SAMPLING:

Quantity Required: 50 mL Container : Plastic

### ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the 5.0 mg/L as Si level.

N.B. Chloride is determined simultaneously.

#### INSTRUMENTION:

Boxed-FIA system consisting of basic automated modular continuous flow system plus the following modules: sample injection valve with air-flow controls, timer, bubble-gate. Colourimetric measurement is through a 5.0 cm. light path at 660 nm. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.02

Detection Criterion (T): 0.04

### CALIBRATION:

BL plus 10 standards

### CONTROLS:

Calibration: LTBL plus 3 standards,eg,QCA, QCB/C, QCD

Drift : BL plus 4 standards

### MODIFICATIONS:

04/07/83 - Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10. The analytical rate was tripled. Concentrations of QC standards adjusted accordingly.

27/03/85 - Silicon analytical range was changed from 0-5.00mg/L to 0-10.00mg/L. First three months' data were omitted because they were not comparable with the later ones.

### NOTES:

Calibration standard is a hydrate: Na2SiO3.9H2O

## SILICON - REACTIVE SILICATES QUALITY CONTROL DATA FROM 27/03/85 TO 30/12/85

ab: Rivers and Lakes

Analytical Range: 0.04 to 10.00 mg/L as Si

ALIBRATION CON	KOL	
----------------	-----	--

a : b : a+b : a-b : c :	Data Concn 54 6.00 54 1.60	Measured 6.02 0.0 1.63 0.0	
b: a+b: a-b: c:	54 1.60	1.63	
b: a+b: a-b: c:	54 1.60	1.63	
a+b : a-b : c :	The second secon	그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그	03 0.041
a-b : c :			
c :	54 7.60	7.65 0.6	Ø5 Ø.13Ø
	54 4.40	4.38 -0.0	02 0.122
d :	53 1.60	1.62 0.6	Ø2 <b>Ø.</b> Ø26
	53 0.40	0.41 0.0	01 0.013
c+d :	53 2.00	2.03 0.0	03 0.031
c-d :		1.21 0.0	01 0.027

.d.(AB): Sw(within run): 0.086 S(between runs): 0.089 S/Sw: 1.03 .d.(CD): Sw(within run): 0.019 S(between runs): 0.021 S/Sw: 1.08

n any given day the calibration is accepted if the values obtained lie within ne ranges:

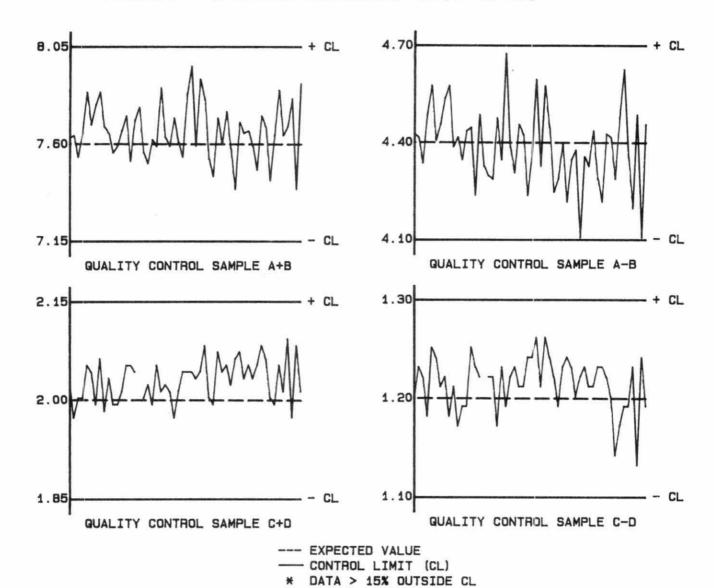
7.15 to 8.05 for A+B 4.10 to 4.70 for A-B 1.85 to 2.15 for C+D 1.10 to 1.30 for C-D

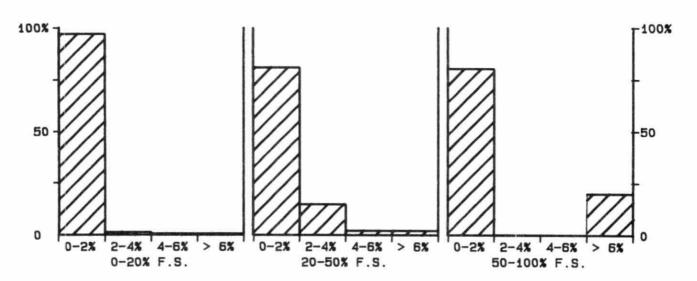
JPL I CATES:	Number of	Sampl	e	Mean(2)	Coefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	62	0.00 -	0.50	0.012	6.2
	37	0.50 -	1.00	0.096	13.1
	54	1.00 -	2.50	0.117	6.9
	31	2.50 -	5.00	0.109	3.3
	5	5.00 -	10.00	0.232	3.6
	189	Overa	11	0.095	N/A

THER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal.	z	54	133	24.3
Long Term Blank	:	54	0.00	0.000

# QUALITY CONTROL GRAPHS SILICON - REACTIVE SILICATES (MG/L AS SI)

FROM: 27/03/85 TO: 30/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 10 MG/L AS SI

### *** SODIUM ***

DENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '74

LIS Test Name Code: NAUR Units : mg/L as Na Work Station Code: WNAK Unit Code : 064811

Method Code : 002BA1 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

AMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

NALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Potassium

s added as a suppressant via an automated sampling train.

Approximate absorbance: 0.16 at the 50 mg/L level.

NSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical

anges are obtained from the output of the AAS.

EPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.1

Detection Criterion (T):0.7

ALIBRATION:

BL plus 2 standards

ONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Orift : BL plus 3 standards

### SODIUM QUALITY CONTROL DATA FROM 02/01/85 TO 31/12/85

Lab: Domestic Water

Analytical Range: 0.7 to 200 mg/L as Na

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	2	98	130	129	-1	1.4
ь	1	98	33	33	0	0.7
a+b	1	98	163	162	-1	1.5
a-b	1	98	97	96	<b>-1</b>	1.5
c	1	98	32.5	32.8	0.3	0.47
d	1	98	6.5	6.7	0.2	0.26
c+d	1	98	39.0	39.4	0.4	0.60
c - <b>d</b>	1	98	26.0	26.1	0.1	0.47

s.d.(AB): Sw(within run): 1.1 S(between runs): 1.1 S/Sw: 1.04 s.d.(CD): Sw(within run): 0.33 S(between runs): 0.38 S/Sw: 1.14

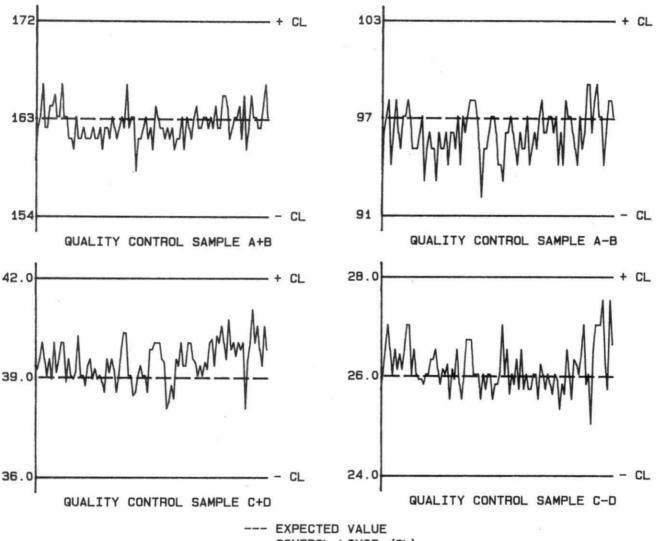
On any given day the calibration is accepted if the values obtained lie within the ranges:

154 to 172 for A+B 91 to 103 for A-B 36.0 to 42.0 for C+D 24.0 to 28.0 for C-D

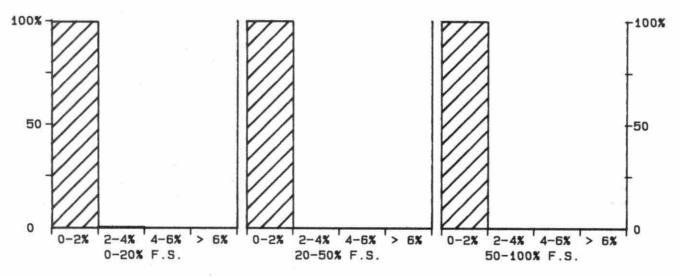
DUPLICATES	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn	Span	s.d.	of var.(%)
	108	0.0 -	10.0	0.23	5.1
	61	10.0 -	20.0	0.29	2.1
	54	20 -	50	0.7	2.2
	31	50 -	100	0.8	1.2
	15	100 -	200	1.0	0.7
	269	Over	a11	0.5	N/A

### QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 02/01/85 TO: 31/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS NA

### *** SODIUM ***

#### IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 18/05/79
LIS Test Name Code: NAUR Units : mg/L as Na
Work Station Code: PRAA Unit Code : 064811
Method Code : 002EA1 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

### SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

### ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 589.0 nm with an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train. Approximate absorbance: 0.5 at the 1.00 mg/L level.

### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer(AAS) system

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.005

Detection Criterion (T): 0.016

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

### MODIFICATIONS:

17/05/85 - Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of a microcomputer to co-ordinate sampler, injection, AAS "read", and data reduction. Sample required reduced to  $5\mbox{mL}.$ 

## SODIUM QUALITY CONTROL DATA FROM 03/01/85 TO 20/12/85

.ab: Precipitation

Analytical Range: 0.016 to 1.00 mg/L as Na

10'	IBRAT	LACOR	CON	וחכודיו	
	IDKU	LUIN	LUN	I PELSE.	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	2	75	0.600	0.605	0.005	0.0090
ь	1	77	0.100	0.102	0.002	0.0099
a+b	:	75	0.700	0.707	0.007	0.0149
a-b	:	75	0.500	0.503	0.003	0.0113

.d.(AB): Sw(within run): 0.0080 S(between runs): 0.0095 S/Sw: 1.18

In any given day the calibration is accepted if the values obtained lie within he ranges:

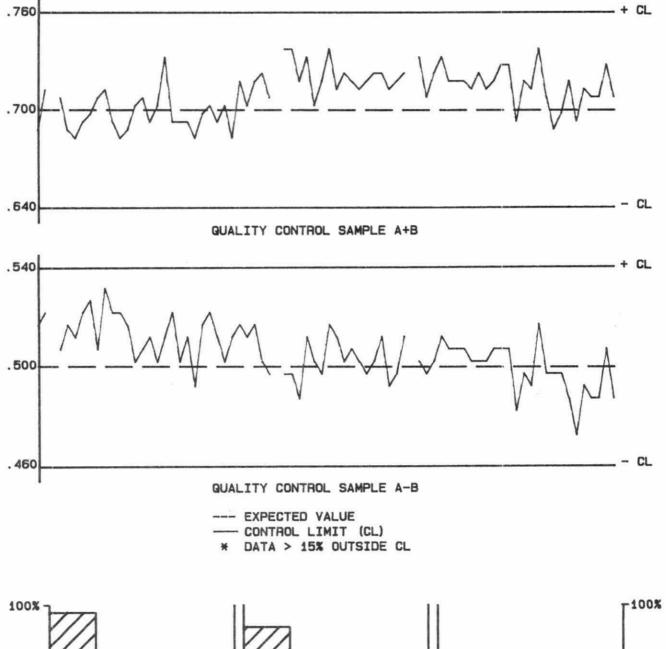
0.640 to 0.760 for A+B 0.460 to 0.540 for A-B

UPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	123	0.000 - 0.100	0.0053	12.6
	20	0.100 - 0.200	0.0086	6.7
	30	0.20 - 1.00	0.010	2.1
	173	Overal1	0.007	N/A

ETECTION CRITERION: 0.016

### QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 03/01/85 TO: 20/12/85



-50 50 2-4% 0-2% 2-4% 4-6% > 6% 0-2% 4-6% > 6% 0-2% 2-4% 4-6% 20-50% F.S. 50-100% F.S. 0-20% F.S.

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 1 MG/L AS NA

#### *** SODIUM ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74 LIS Test Name Code: NAUR Units : mg/L as Na Work Station Code : RMAAS Unit Code : 064811 Method Code : 002CA1,002DA1 Supervisor : J. Crowther Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: RMAAS : 1,07

#### INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01 CALIBRATION:

Detection Criterion (T):0.08*,0.20,0.08

BL plus 10 standards

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA for each analytical range : BL plus 1 standard for each analytical range Drift

#### MODIFICATIONS:

01/12/81- Calibration range became 10.0 mg/L full scale; second analytical range was dropped.

01/03/84- Analytical range(RMNAKL) was added; full scale:2.00 mg/L. This range is currently restricted to special programs.

01/09/84- Analytical range(RMNAKH) was increased from 10.0 to 20.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Potassium is no longer determined simultaneously. 25/09/85- Calibration range remains at 20.0 mg/L full scale but second analytical range was dropped. Concentrations of QC standards were adjusted accordingly. Microcomputer controlled system.

#### NOTES:

Three analytical ranges were used during 1985: 2.00, 20.0, and 20.0 mg/L full scale as Na. Corresponding detection criteria are given above.

*T value is based on duplicate analyses at concentrations above the lowest range.

## SODIUM QUALITY CONTROL DATA FROM 03/01/85 TO 09/09/85

Lab: Rivers and Lakes

Analytical Range: 0.20 to 20.00 mg/L as Na

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	37	15.00	15.12	0.12	0.318
b	:	37	5.00	5.04	0.04	0.214
a+b	:	37	20.00	20.16	0.16	0.437
a-b	:	37	10.00	10.07	0.07	0.320

s.d.(AB): Sw(within run): 0.226 S(between runs): 0.271 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

19.10 to 20.90 for A+B 9.40 to 10.60 for A-B

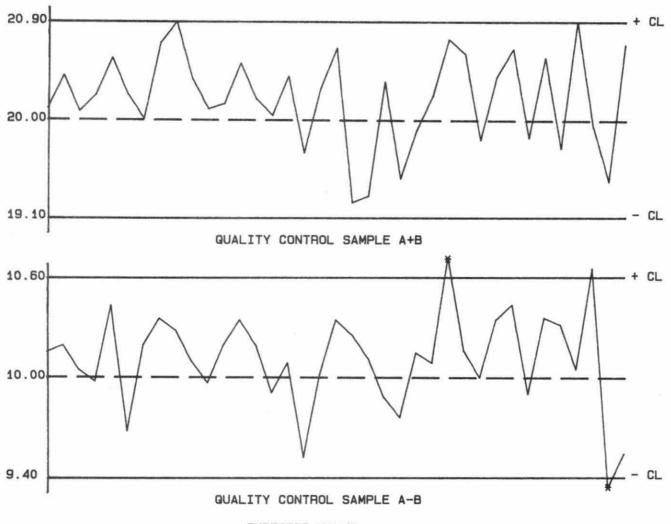
DUPL ICATES:	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn S	Span	s.d.	of var.(%)
	45	0.00 -	1.00	0.068	12.1
	15	1.00 -	2.00	0.088	5.8
	14	2.00 -	5.00	0.144	5.0
	9	5.00 -	10.00	0.121	1.8
	8	10.00 -	20.00	0.379	2.3
	91	Overa	a 1 1	0.144	N/A

#### DETECTION CRITERION: 0.20

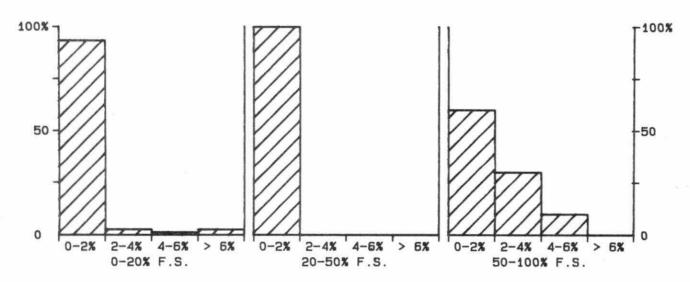
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	30	1.01	0.100
Long Term Blank	:	0	N/A	N/A

### QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 03/01/85 TO: 09/09/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 20 MG/L AS NA

## SODIUM QUALITY CONTROL DATA FROM 02/10/85 TO 30/12/85

Lab: Rivers and Lakes

Analytical Range: 0.08 to 20.00 mg/L as Na

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		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.		23	16.00	16.00	0.00	0.301
b	:	23	1.40	1.40	0.00	0.038
a+b		23	17.40	17.41	0.01	0.304
a-b	:	23	14.60	14.60	0.00	0.302

s.d.(AB): Sw(within run): 0.214 S(between runs): 0.215 S/Sw: 1.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

16.50 to 18.30 for A+B 14.00 to 15.20 for A-B

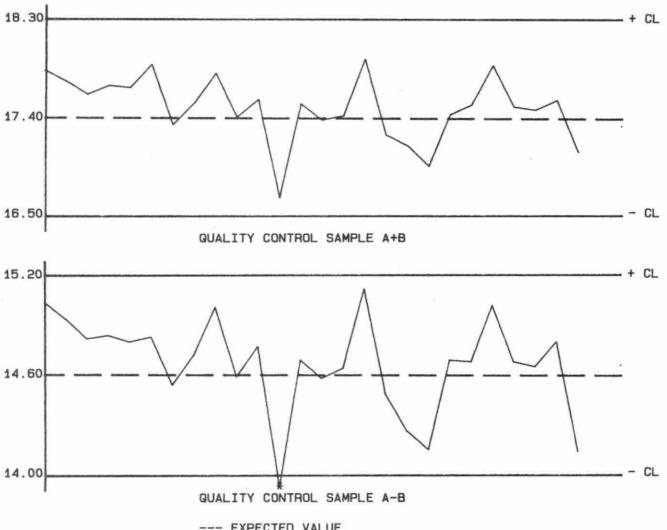
DUPLICATES:	Number of Data Pairs	Samp! Concn S		Mean(2) s.d.	Coefficient of var.(%)
	44	0.00 -	1.00	0.026	3.9
	16	1.00 -	2.00	0.051	3.6
	6	2.00 -	4.00	0.092	3.0
	3	4.00 -	10.00	0.417	7.2
	1	10.00 -	20.00	N/A	N/A
	70	Overa	al 1	0.116	N/A

#### DETECTION CRITERION: 0.08

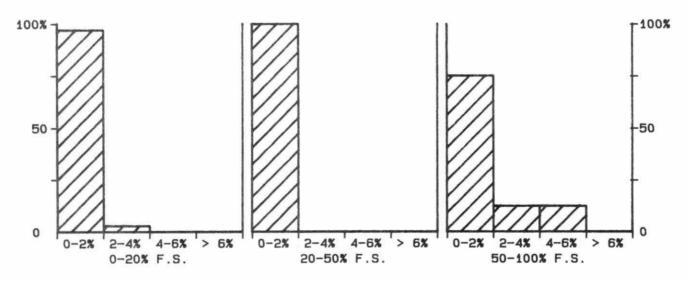
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Absorbance	:	Ø	N/A	N/A
Long Term Blank	:	0	N/A	N/A

### QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 25/09/85 TO: 30/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS NA

#### SODIUM QUALITY CONTROL DATA FROM 07/01/85 TO 10/09/85

Lab: Rivers and Lakes

Analytical Range: N/A to 2.00 mg/L as Na

CAL.	BRAT	MOL	CON	TROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	1	39	1.50	1.50	0.00	0.027
ь		39	0.50	0.50	-0.00	0.023
a+b		39	2.00	2.00	-0.00	0.039
a-b	E	39	1.00	1.00	0.00	0.032

s.d.(AB): Sw(within run): 0.023 S(between runs): 0.025 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B 0.94 to 1.06 for A-B

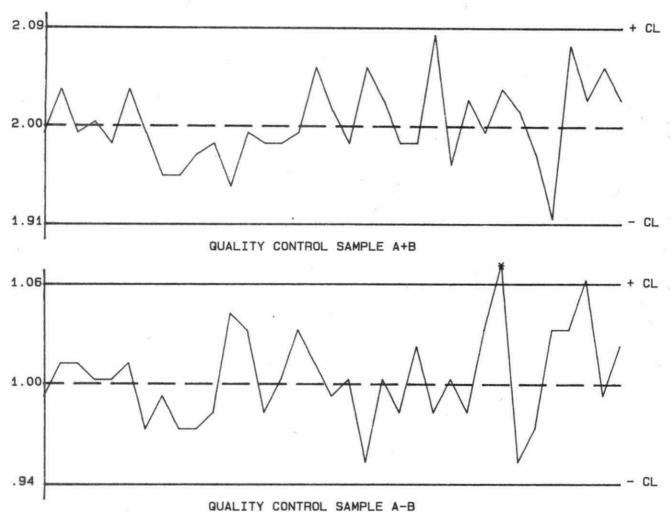
DUPL I CATES	Number of	Sampl	e	Mean(2)	Coefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	1	0.00 -	0.20	N/A	N/A
	15	0.20 -	0.50	0.026	6.0
	66	0.50 -	1.00	0.037	5.3
	23	1.00 -	2.00	0.057	4.1
	105	Overa	11	0.041	N/A

#### DETECTION CRITERION: N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	33	0.96	0.084
Long Term Blank		0	N/A	N/A

# QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

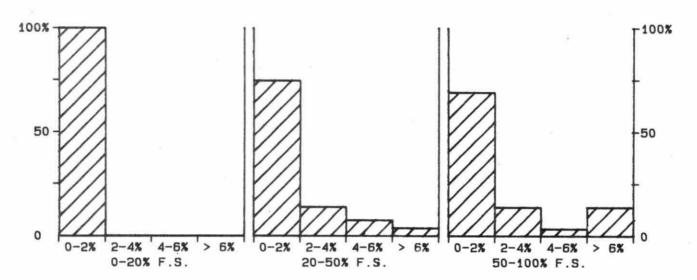
FROM: 07/01/85 TO: 10/09/85



--- EXPECTED VALUE

- CONTROL LIMIT (CL)

* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 MG/L AS NA

#### *** SOLIDS - DISSOLVED ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74

LIS Test Name Code: RSF Units : mg/L Work Station Code: RMTSD Unit Code : 064000

Method Code : 101A15 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sample (shaken) is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50.0 mL of filtrate is pipetted into a preweighed ceramic dish, dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5-decimal places), drying oven, suction filtration apparatus, ceramic dishes

-Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.5 Detection Criterion (T): Not applicable*

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA

Recovery : LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked frequently

#### MODIFICATIONS:

15/01/82- QC program was expanded to include recovery standards

01/05/84- Microcomputer control was introduced

#### NOTES:

*Dissolved solids in surface waters normally are estimated when the conductivity of the sample is less than 400 uS/cm:

Dissolved solids(mg/L) = 0.65 x Conductivity(uS/cm)

Hence, few data from direct measurements at low concentrations are available tocalculate the detection criterion of this gravimetric test.

## SOLIDS - DISSOLVED QUALITY CONTROL DATA FROM 13/02/85 TO 31/12/85

Lab: Rivers and Lakes

Analytical Range: N/A to 1000 mg/L

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		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		153	50.0000	50.0002	0.0002	0.00020
b	:	153	10.0000	10.0001	0.0001	0.00008
a+b		153	60.0000	60.0002	0.0002	0.00024
a-b	:	153	40.0000	40.0001	0.0001	0.00018

s.d.(AB): Sw(within run): 0.00013 S(between runs): 0.00015 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

59.998 to 60.001 for A+B 39.998 to 40.001 for A-B

RECOVER IES:		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
44,					
r1	:	116	800	800	15.5
r2	1	116	300	305	14.4

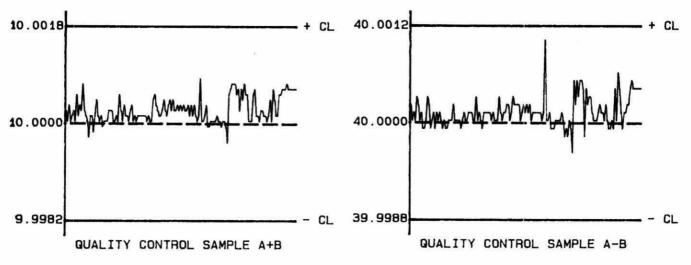
DUPLICATES:	Number of	S	a.mp	le	Mean(2)	Coefficient
	Data Pairs	Con	Concn Span		s.d.	of var.(%)
	2	Ø	-	50	3.6	7.9
	3	50	-	100	5.2	8.2
	23	100	-	250	8.4	4.4
	164	250	-	500	11.6	3.3
	25	500	-	1000	9.2	1.4
	217	0	vera	all	10.9	N/A

DETECTION CRITERION: N/A

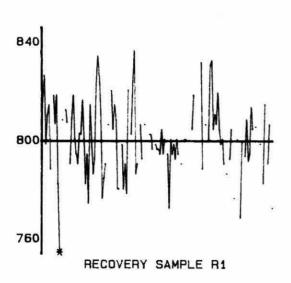
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Blank	58	1.61	7.513

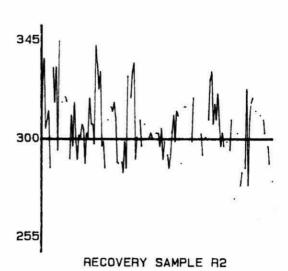
# QUALITY CONTROL GRAPHS SOLIDS - DISSOLVED (MG/L)

FROM: 13/02/85 TO: 31/12/85

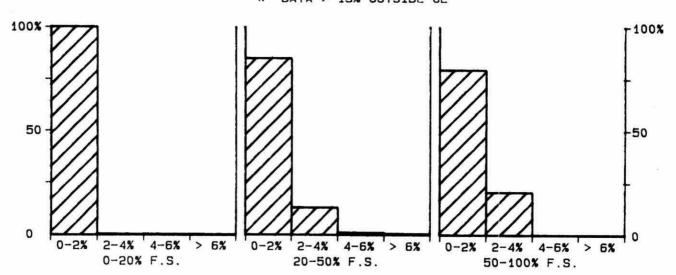


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 1000 MG/L

*** SOLIDS - DISSOLVED ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61

LIS Test Name Code: RSF Units : mg/L Work Station Code: SOLIDS Unit Code : 064000

Method Code : 101AI5 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

#### SAMPLING:

Quantity Required: 125 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50 or 100 mL of filtrate is pipetted into a preweighed Teflon dish, dried at 103 to 105 C, and stored in a dessicator until cool. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5-decimal places), drying oven, suction filtration apparatus, Teflon dishes

Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W): 0.5 Detection Criterion (T): 9.2

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA Recovery: LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked at least 4 times daily

#### MODIFICATIONS:

15/01/82- Microcomputer control was introduced

#### NOTES:

As the same two balances are used for all solids analyses in the Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report.

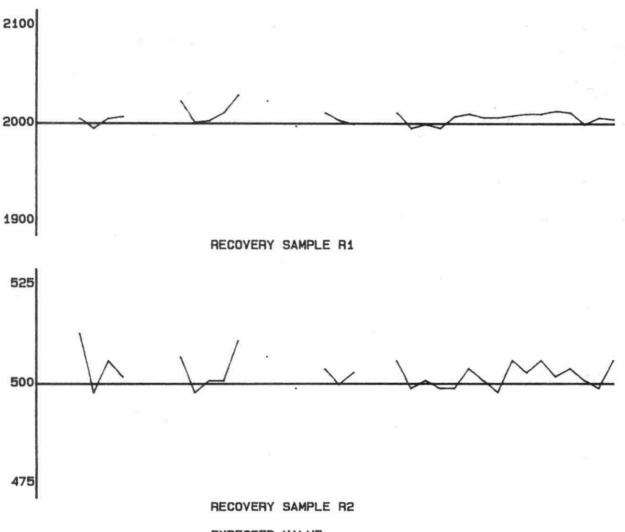
No data summary is available for period not covered in performance report.

## SOLIDS - DISSOLVED QUALITY CONTROL DATA FROM 01/02/85 TO 18/12/85

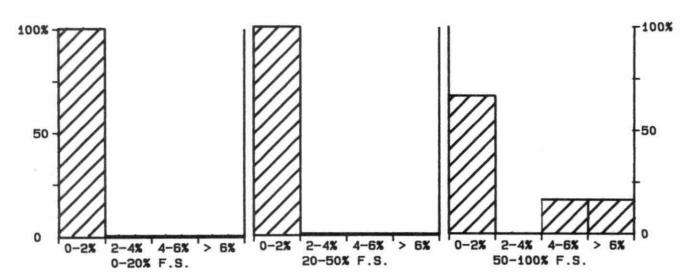
Lab: Sewage an	d Industrial	Waste	Analytical Ran	ge: 9.2 to 3000	mg/L
RECOVERIES	Numbe of Dat		Av.Conc. Measured	Standard(1) Deviation	
r1	: 30	2000	2004	8.2	
20.00		1.7 m; 5-2.0 m; 1-0.0		( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	
re	: 30	500	502	3.8	
DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient	
	Data Pairs	Concn Span	s.d.	of var.(%)	
	5	0.0 - 200.0	3.06	2.0	
	11	200 - 400	4.8	1.7	
	5	400 - 600	7.5	1.6	
	7	600 - 1000	4.7	0.6	
	1	1000 - 3000	N/A	N/A	
	29	Overall	6.5	N/A	
DETECTION CRIT	ERION: 9.2				
OTHER CHECKS:		Number	Data	Standard(1)	
		of Data	Mean	Deviation	
B1	anK :	28	0.07	5.959	

### QUALITY CONTROL GRAPHS SOLIDS - DISSOLVED (MG/L)

FROM: 01/02/85 TO: 18/12/85



--- EXPECTED VALUE * DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 3000 MG/L

#### *** SOLIDS - IGNITED ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61 LIS Test Name Code: RSFA,RSPA,RSTA Units : mg/L or mg/kg

Work Station Code : SOLIDS Unit Code : 064000

Method Code : 101AI5,201AI5,001AI5 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachate,

Effluents

#### SAMPLING:

Quantity Required: 75-500 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

The procedure for dissolved, particulate, or total solids is followed and the dried residue is ignited at 600 C for one hour in a muffle furnace. As soon as practical the dish is transferred to a desiccator to cool. The ignited or ash weight is obtained as the difference between the final ignited weight and the original dish weight. Similarly the volume used in the ignited calculations is the volume selected for the original dried solids measurement. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5 decimal places), muffle furnace, ceramic dishes, Petri dishes -Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W): 1,0.1,0.5
Detection Criterion (T): 6,18.6*,52

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 4 S class weights, eg, QCA

Drift : Balance zero is checked at least 4 times daily

#### MODIFICATIONS:

01/05/82 Microcomputer control was introduced

#### NOTES:

-In the order listed above, W and T values refer to the residual ash after ignition of the dried residual from dissolved, particulate, and total solids determinations.

-Duplicate data refer to ash residuals rather than loss on ignition.

-Detection criteria estimates are unreliable due to limited data; samples requiring these tests are usually sewage sludges with high solids contents.

-As the same two balances are used for all solids analyses in the

Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report for Ignited Dissolved and Ignited Total tests, and in the Solids-Particulate report for Ignited Particulate tests.

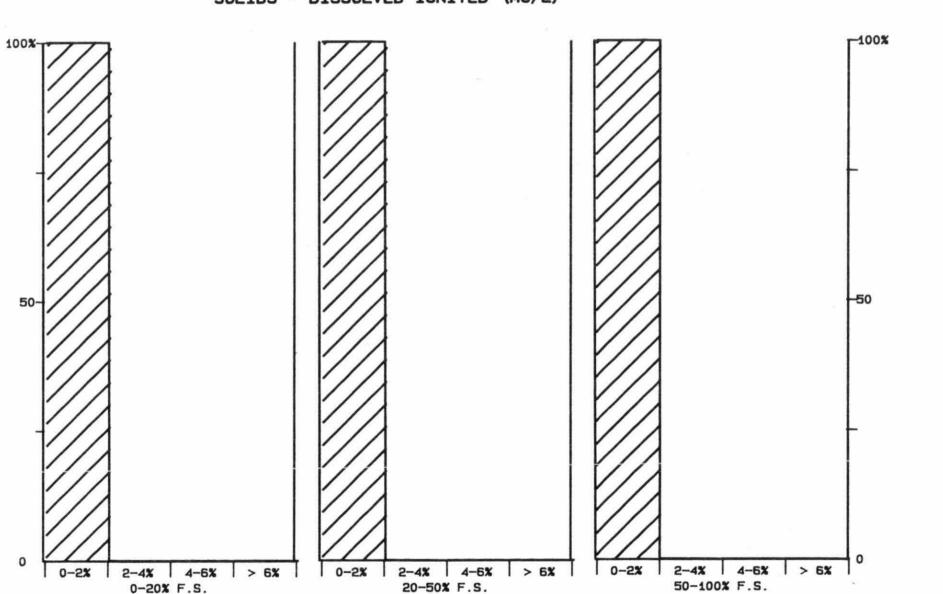
*T value is based on duplicate data above the lowest range.

## SOLIDS - DISSOLVED IGNITED QUALITY CONTROL DATA FROM 07/01/85 TO 18/12/85

ab: Sewage	and Industria	l Waste			Analytical	Range: - to 3000	mg/L
UPLICATES	Number of	S	a.mpl	e	Mean(2)	Coefficient	* 9
	Data Pairs	Cond	cn S	pan	s.d.	of var.(%)	
	8	0	-	200	1.99	1.4	
	4	200	-	400	5.3	1.8	
	5	400	-	600	9.8	2.0	
	10	600	-	1000	9.3	1.2	
	12	1000	- ;	3000	19.9	1.5	
	39	0	vera	1 1	12.6	N/A	
ETECTION C	RITERION: 6.0	*					
THER CHECK	SI	Numbe	er		Data	Standard(1)	
		of Dat	ta		Mean	Deviation	
	Blank :	38			0.52	8.342	

### QUALITY CONTROL GRAPH SOLIDS - DISSOLVED IGNITED (MG/L)

FROM: 07/01/85 TO: 18/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 3000 MG/L

47

## SOLIDS - PARTICULATE IGNITED QUALITY CONTROL DATA FROM 07/01/85 TO 23/12/85

ab: Sewage and Industrial Waste

Analytical Range: - to 3000 mg/L

31.1	BRAT	TON	CON	TROL
-		4011		

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	67	0.50002	0.49999	-0.00003	0.000019
b		67	0.05002	0.04999	-0.00003	0.000015
a+b	:	67	0.55004	0.54998	-0.00006	0.000028
ab	:	67	0.45000	0.45000	-0.00000	0.000019

.d.(AB): Sw(within run):0.000013 S(between runs):0.000017 S/Sw: 1.27

n any given day the calibration is accepted if the values obtained lie within me ranges:

0.5470 to 0.5530 for A+B 0.4480 to 0.4520 for A-B

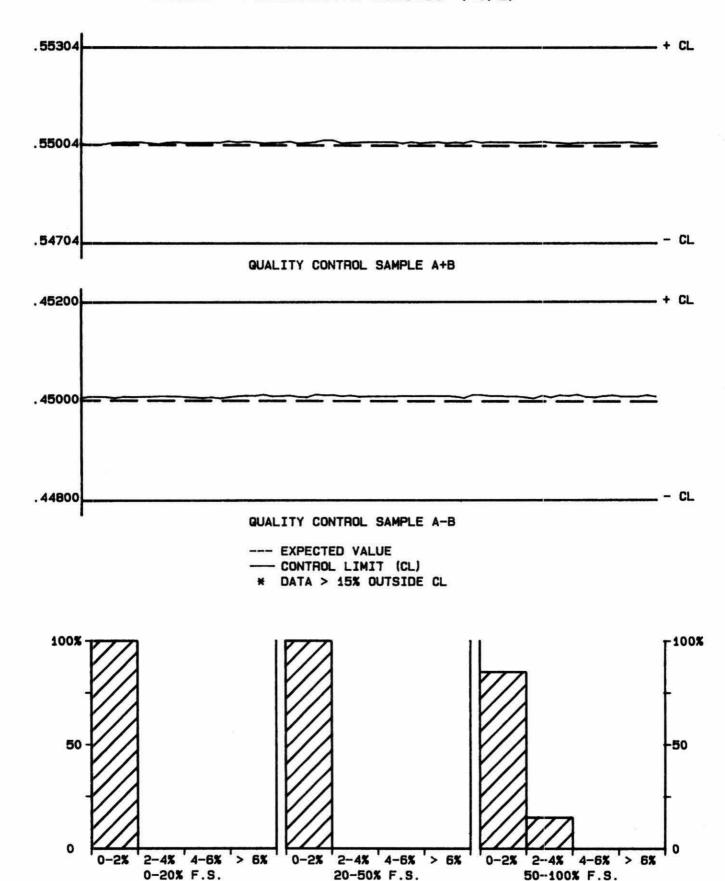
JPL ICATES:	Number of Data Pairs	Sample Concn Span			Mean(2)	Coefficient of var.(%)
	2	0.0	9 -	100.0	0.18	1.1
	8	100	-	500	6.2	1.9
	20	500	-	1000	10.3	1.4
	4	1000	-	1500	11.6	1.0
	15	1500	-	3000	24.4	1.1
	49		Over	a11	15.6	N/A

#### TECTION CRITERION: 0.5

THER CHEC	KS:		Number	Data	Standard(1)
			of Data	Mean	Deviation
	Blank	:	51	-0.61	0.497

# QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE IGNITED (MG/L)

FROM: 07/01/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 3000 MG/L

## SOLIDS - TOTAL IGNITED QUALITY CONTROL DATA FROM 07/01/85 TO 26/11/85

ab. Sewaye	and industria	I Waste		nnalytical	. Kange: - to 30000 m	3/6
UPLICATES:	Number of	Sample		Mean(2)	Coefficient	
	Data Pairs	Concr	Span	s.d.	of var.(%)	
	16	ø -	4000	17.2	0.8	
	5	4000 -	8000	57.6	0.9	
	9	8000 -	12000	90.9	ø.9	
	7	12000 -	18000	115.6	0.7	
	15	18000 -	30000	129.5	0.5	
	52	Ove	rall	92.1	N/A	
ETECTION CR	ITERION: 52					
THER CHECKS	ı	Number		Data	Standard(1)	
		of Data	i,	Mean	Deviation	
			•6			

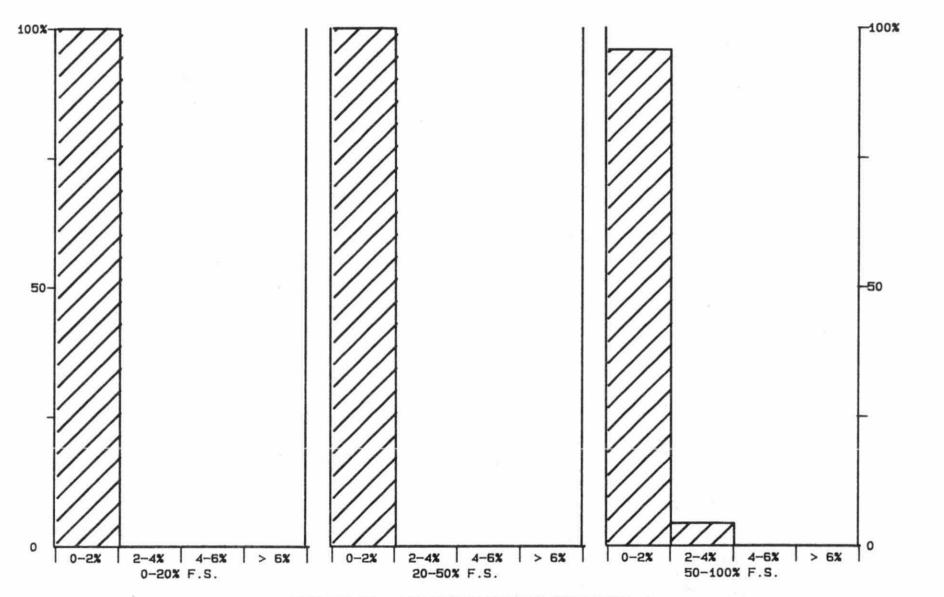
0.62

6.887

Blank

#### FROM: 07/01/85 TO: 26/11/85

# QUALITY CONTROL GRAPH SOLIDS - TOTAL IGNITED (MG/L)



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 30000 MG/L

*** SOLIDS - PARTICULATE ***

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74

LIS Test Name Code: RSP Units : mg/L Work Station Code: RMTSD Unit Code : 064000

Method Code : 202A16 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

#### SAMPLING:

Quantity Required: 150-500 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

An appropriate shaken sample volume (100 to 500 mL) is quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a prewashed and preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 25 mL distilled water; the rinse step is repeated. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(6-decimal places), drying oven, suction filtration apparatus

-Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 1.4

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA Recovery: LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked frequently

Blank : Filter washed with 50 mL distilled water, result uncorrected.

#### MODIFICATIONS:

01/03/84 -QC program was expanded to include recovery standards.

01/05/84 -Microcomputer control was introduced.

### SOLIDS - PARTICULATE QUALITY CONTROL DATA FROM 02/01/85 TO 31/12/85

Lab! Rivers and Lakes

Blank

Analytical Range: 1.4 to 1000 mg/L

0.445

CALIBRE	HOITE	CONTROL				
		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	174	100.00	99.97	-0.03	0.047
ь	1	174	20.00	19.90	-0.10	0.019
a+b	ŧ	174	120.00	119.87	-0.13	0.066
a-b	1	174	80.00	80.07	0.07	0.030

On any given day the calibration is accepted if the values obtained lie with

s.d.(AB): Sw(within run): 0.021 S(between runs): 0.036 S/Sw: 1.69

On any given day the calibration is accepted if the values obtained lie within the ranges:

119.81 to 120.19 for A+B 79.88 to 80.12 for A-B

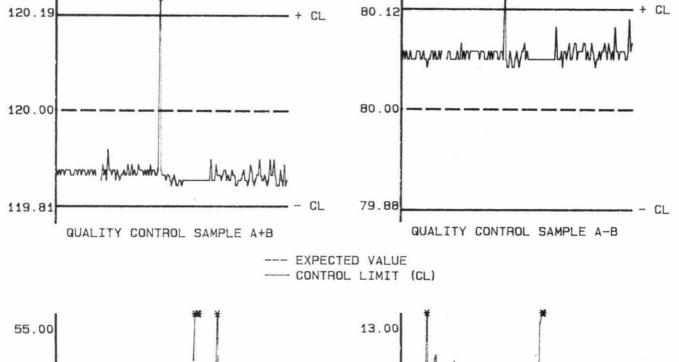
	rise in the				
RECOVER IES:	N	umber E	xpected	Av.Conc.	Standard(1)
	of	Data C	oncn	Measured	Deviation
r 1	: 13	37	50.00	48.87	2.999
r2	1 13	35	10.00	9.70	1.442
DUPL ICATES:	Number of	f Sam	ple	Mean(2)	Coefficient
	Data Pair	s Concn	Span	s.d.	of var.(%)
	96	0.0 -	5.0	0.46	17.0
	57	5.0 -	10.0	0.85	11.5
	55	10.0 -	25.0	1.35	8.6
	63	25.0 -	100.0	3.06	7.0
	12	100 -	1000	6.6	2.0
	283	Ove	rall	2.1	N/A
DETECTION CRI	TERION	1.4			
OTHER CHECKS:		Number		Data	Standard(1)
		of Data		Mean	Deviation

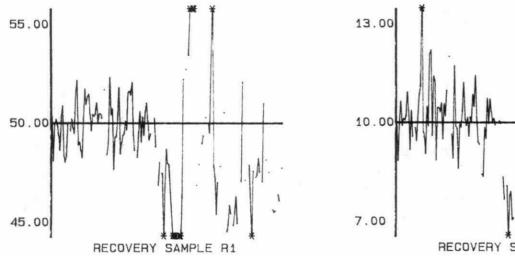
-0.54

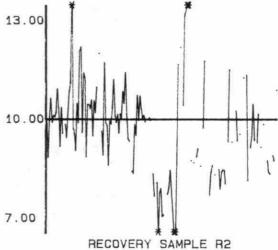
63

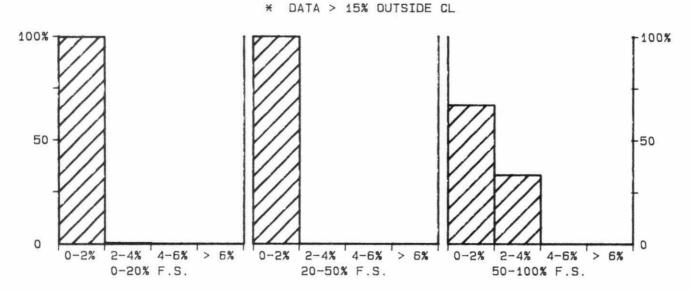
### QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE (MG/L)

FROM: 02/01/85 TO: 31/12/85









CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 MG/L

#### *** SOLIDS - PARTICULATE ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61

LIS Test Name Code: RSP Units : mg/L Work Station Code: SOLIDS Unit Code : 064

Method Code : 201AI5 Supervisor : P. Campbell

Sample Type/Matrix: Sewage, Industrial Waste, Drinking Waters, Leachate, Effluents

SAMPLING:

Quantity Required: 75-500 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

An appropriate sample volume(25 to 500 mL) is quickly poured into a grduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 50 mL distilled water. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(5-decimal places), drying oven, suction filtration apparatus

-Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.1

Detection Criterion (T): 2.5

#### CALIBRATION:

Balance zero

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA for each balance (results in grams)

Recovery : LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked at least 4 times daily

Blank : Filter rinsed with 50 mL distilled water and corrected using

blank correction factor outlined below (expected result is 0.00 mg/L).

#### MODIFICATIONS:

01/07/81- Current microcomputer control system was introduced.

01/03/83- QC program was expanded to include recovery standards.

01/05/83 - Prerinsing of filters was discontinued. Instead, 5 filters from each box of 100 are weighed before and after rinsing to correct results for filters used with samples.

01/07/83 - New glass and acrylic filter holders (Whatman 90 mm) replaced Buchner funnels. Size and position of pores in the two types of holders is similar, but the filtration area is smaller and the seal at the filter edge is superior with the new holder.

## SOLIDS - PARTICULATE QUALITY CONTROL DATA FROM 02/01/85 TO 24/12/85

ab: Sewage and Industrial Waste Analytical

Analytical Range: 2.5 to 3000 mg/L

01	TODAT	TON	COLD	. 100
пь	IBRAT	TON	CUNI	KUL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	90	0.50002	0.50002	-0.00000	0.000026
ь	:	90	0.05002	0.05002	-0.00000	0.000025
a+b	:	90	0.55004	0.55004	-0.00000	0.000046
a-b	:	90	0.45000	0.45000	0.00000	0.000022

.d.(AB): Sw(within run):0.000016 S(between runs):0.000026 S/Sw: 1.64

n any given day the calibration is accepted if the values obtained lie within he ranges:

0.5470 to 0.5530 for A+B 0.4479 to 0.4520 for A-B

ECOVER IES:		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r1		74	187	182	2.1
r2	:	67	47.2	47.2	1.28

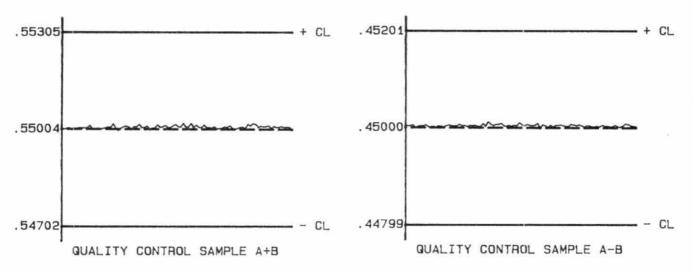
UPLICATES:	Number of	Sample	Mean(2)	Coefficient	
	Data Pairs	Concn Span	s.d.	of var.(%)	
	15	0.0 - 25.0	0.83	6.8	
	24	25.0 - 50.0	2.92	7.4	
	78	50.0 - 150.0	2.29	2.4	
	46	150 - 3000	18.0	2.2	
	163	Overal1	9.8	N/A	

#### ETECTION CRITERION: 2.5

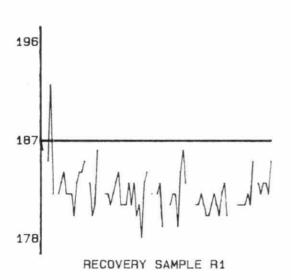
THER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Blank	:	83	0.06	0.357

### QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE (MG/L)

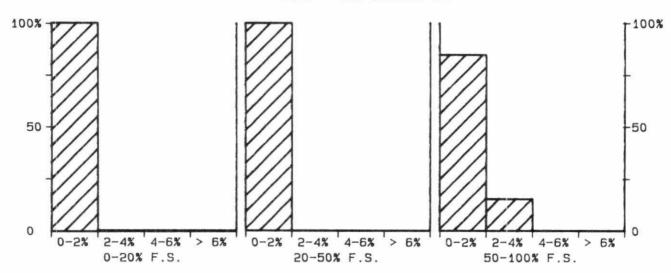
FROM: 02/01/85 TO: 24/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 3000 MG/L

#### *** SOLIDS- TOTAL ***

#### IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: RST Units : mg/L or mg/K

LIS Test Name Code: RST Units : mg/L or mg/kg Work Station Code: SOLIDS Unit Code : 064000

Method Code : 001AI5 Supervisor : P. Campbell

Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents, Sludge

#### SAMPLING:

Quantity Required: 75 mL to 125 mL Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

A 50.0 mL aliquot of sample is pipetted into a preweighed teflon dish, dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing the total residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

- -Balance(4/5-decimal places), drying oven, ceramic dishes
- -Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.5

Detection Criterion (T): 13

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration : 2 S class weights, eg, QCA (results in grams)

Recovery : BL plus 2 standards, eg, R1

Drift : Balance zero is checked at least 4 times daily

#### MODIFICATIONS:

15/01/82 Microcomputer control was introduced

## SOLIDS - TOTAL QUALITY CONTROL DATA FROM 07/01/85 TO 23/12/85

Lab: Sewage and Industrial Waste

Blank

Analytical Range: 13 to 60000 mg/L

CALI	BRAT	ION	CONTROL:	
------	------	-----	----------	--

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	70	50.0004	50.0004	0.0000	0.00011
ь	:	70	30.0001	30.0001	-0.0000	0.00009
a.+b	:	70	80.0005	80.0005	-0.0000	0.00018
a-b	I	70	20.0003	20.0004	0.0001	0.00010

s.d.(AB): Sw(within run): 0.00007 S(between runs): 0.00010 S/Sw: 1.42

On any given day the calibration is accepted if the values obtained lie within the ranges:

79.999 to 80.002 for A+B 19.999 to 20.001 for A-B

0.28

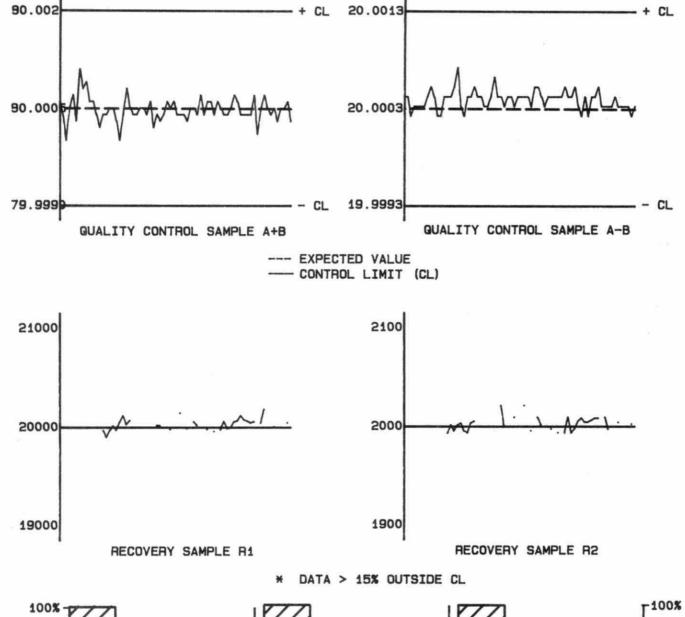
3.965

RECOVER IES:		Numb	••	F.,		A C	04
KECOVEK IES.				Expected		Av.Conc.	Standard(1)
		of Da	ta	Co	ncn	Measured	Deviation
			-				
r1	:	33		20	000	20016	58.5
re	:	33		2	999	2001	7.3
DUPLICATES	Numbe	r of	s	amp	l e	Mean(2)	Coefficient
	Data	Pairs	Con	cn	Span	s.d.	of var.(%)
	11		Ø	-	2000	4.4	0.8
	8	E	2000	-	10000	50.8	0.8
	10		10000	-	20000	80.2	0.6
	12	}	20000	-	40000	368.5	1.2
	10		40000	-	60000	345.6	0.7
	51		0	ver	all	238.8	N/A
DETECTION CRI	TERION	13					
OTHER CHECKS:			Numb of Da			Data Mean	Standard(1) Deviation

34

### QUALITY CONTROL GRAPHS SOLIDS - TOTAL (MG/L)

FROM: 07/01/85 TO: 23/12/85



100% 50 0 -2% 2-4% 4-6% > 6% 0-2% 2-4% 4-6% > 6% 0-2% 2-4% 4-6% > 6% 0-20% F.S.

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 60000 MG/L

#### *** SULPHATE ***

#### IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '75 LIS Test Name Code: SSO4UR Units : mg/L as SO4

Work Station Code: WSO4 Unit Code: 064941
Method Code: 003AC1 Supervisor: M. Rawlings

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sample is pumped through an ion exchange column to suppress cationic interferences, and then the sulphate reacts with barium methyl-thymol blue to produce barium sulphate and methyl-thymol blue (MTB). The absorbance of MTB provides a measurement of the sulphate concentration. Approximate absorbance: 1.0 at the 100 mg/L level.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the modules: 37 C heating bath (7.7 mL delay), cationic exchange column. Colourimetric measurement is through a 5.0 cm light path at 460 nm.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.5

Detection Criterion (T): 1.1

#### CALIBRATION:

BL plus 1 standard in duplicate

#### CONTROLS:

Calibration : 2 standards,eg, QCA
Drift : BL plus 2 standards

Interference: BL spiked with 50 mg/L magnesium SO4 and 200 mg/L calcium confirms efficiency of cation removal.

#### MODIFICATIONS:

01/10/81- A carbon dioxide trap was installed on the sodium hydroxide reagent. 01/10/84- A heating bath module (37 C) was installed preceding the colourimeter.

#### SULPHATE QUALITY CONTROL DATA FROM 04/01/85 TO 30/12/85

Lab: Domestic Water

Analytical Range: 1.1 to 100.0 mg/L as SO4

 		The same of the sa	the second secon	
TODET	TONI	COL	TROL.	
LORGI	F F 7 1 7 4	1.1113	I PULLUE - A	

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	89	58.0	58.2	0.2	1.32
b	:	89	11.6	11.9	0.3	0.55
a+b	:	89	69.6	70.1	0.5	1.73
a-b	1	89	46.4	46.2	-0.2	1.04

s.d.(AB): Sw(within run): 0.74 S(between runs): 1.01 S/Sw: 1.37

On any given day the calibration is accepted if the values obtained lie within the ranges:

65.1 to 74.1 for A+B 43.4 to 49.4 for A-B

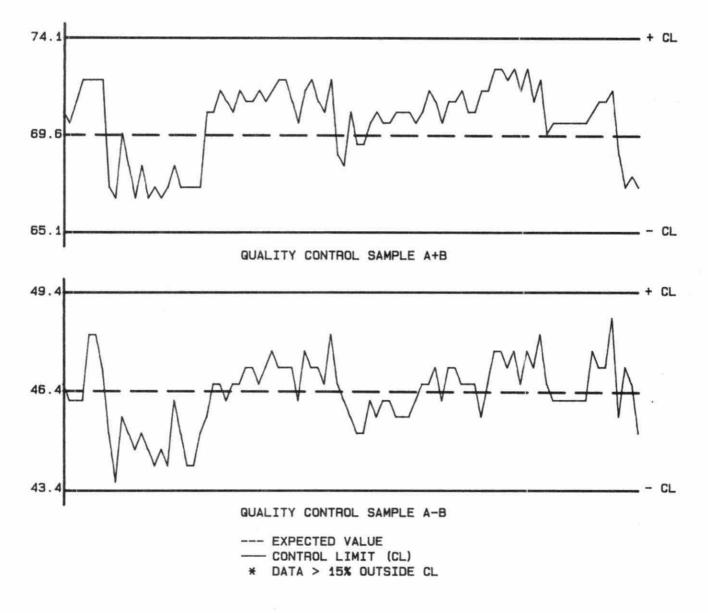
DUPL ICATES:	Number of	Sample	2	Mean(2)	Coefficient
	Data Pairs	Concn S	an	s.d.	of var.(%)
	29	0.0 -	10.0	0.38	6.5
	66	10.0 -	20.0	0.55	3.6
	90	20.0 -	50.0	0.54	1.6
	39	50.0 -	100.0	0.81	1.2
	224	Overal	11	0.58	N/A

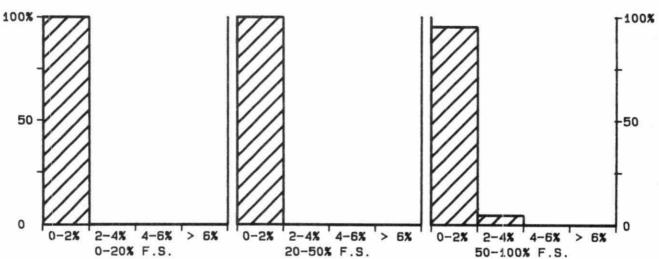
#### DETECTION CRITERION: 1.1

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
50mgS04+200mgCa/L	:	89	50.1	0.57

### QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 04/01/85 TO: 30/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS SO4

#### *** SULPHATE - PRECIPITATION ***

#### IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/04/78
LIS Test Name Code: SSO4UR Units : mg/L as SO4
Work Station Code : PRIC1 Unit Code : 064941
Method Code : 003AI0 Supervisor : M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 10 uS/cm.

Nitrate and chloride are determined simultaneously.

#### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

#### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.09

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

### SULPHATE QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Precipitation

Analytical Range: 0.09 to 10.00 mg/L as SO4

	CONTROL	

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	143	8.00	8.01	0.01	0.058
b		142	2.00	2.00	0.00	0.033
a+b	1	142	10.00	10.01	0.01	0.069
a-b		142	6.00	6.01	0.01	0.065

s.d.(AB): Sw(within run): 0.046 S(between runs): 0.047 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

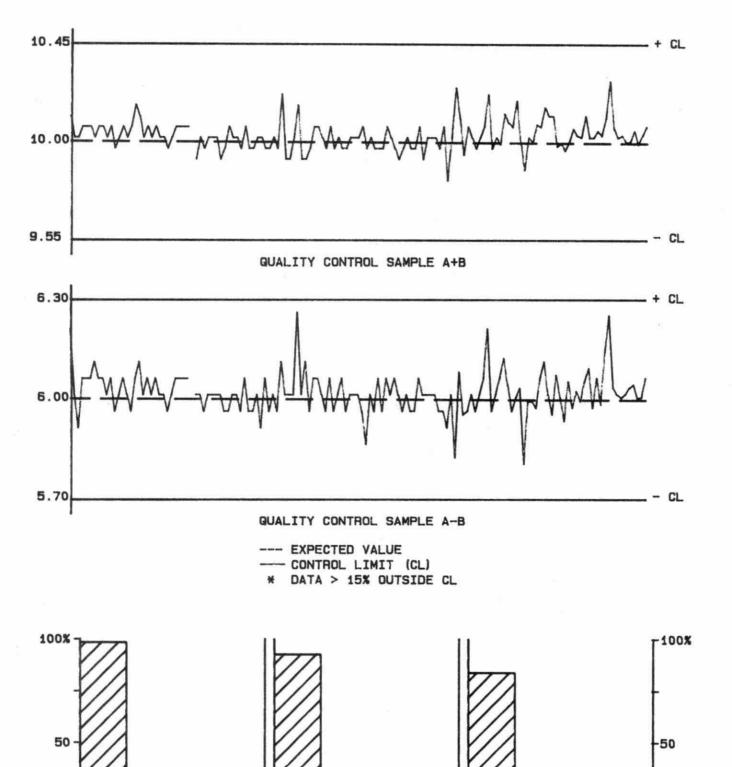
9.55 to 10.45 for A+B 5.70 to 6.30 for A-B

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	25	0.00 - 1.00	0.032	4.7
	40	1.00 - 2.00	0.040	2.5
	68	2.00 - 5.00	0.108	3.1
	47	5.00 - 10.00	0.259	3.7
	180	Overall	0.150	N/A

DETECTION CRITERION: 0.09

# QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 02/01/85 TO: 23/12/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS S04

20-50% F.S.

2-4%

4-6%

> 6%

2-4%

50-100% F.S.

4-6% > 6%

0-2%

> 6%

0-2%

2-4% 4-6%

0-20% F.S.

### *** SULPHATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80

LIS Test Name Code: SSO4FR SSO4NF Units : ug/Filter as SO4

Work Station Code : PRSEQ Unit Code : 361941
Method Code : 004AI0 Supervisor : M. Rawlings

Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and nylon filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bag

SAMPLE PREPARATION:

Filters are extracted with 25 ml. of DDW (teflon) or 25 ml. of .03N NaOH (nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO4.

Full scale conductivity : 30 uS/cm.

Nitrate and chloride are determined simultaneously.

### INSTRUMENTATION:

-Ultrasonic bath; polystyrene tubes

-Automated modular continuous flow ion chromatographic system.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 1.25

Detection Criterion (T): 3

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : 1 standard every 10 samples

### MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified for analysis of teflon and nylon filter extracts by developing the above filter extraction procedure

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

10/05/85 - Microcomputer used for data reduction.

- Three additional calibration standards were set up.

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

### SULPHATE QUALITY CONTROL DATA FROM 04/01/85 TO 27/12/85

.ab: Precipitation

Analytical Range: 3 to 250 ug/Filter as SO4

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	160	200	201	1	1.6
b	E	160	50	50	Ø	0.8
a+b	:	160	250	250	Ø	1.9
a-b	:	160	150	151	1	1.7

i.d.(AB): Sw(within run): 1.2 S(between runs):

1.3 S/Sw: 1.05

In any given day the calibration is accepted if the values obtained lie within the ranges:

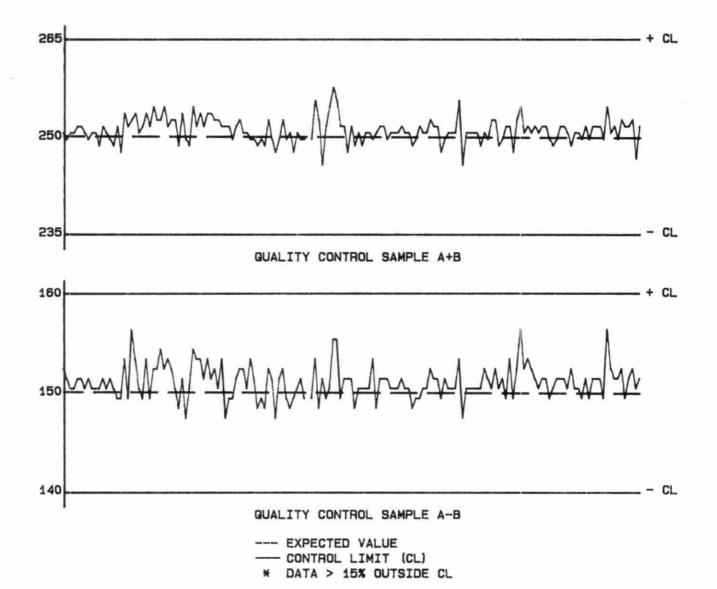
> 235 to 265 for A+B 140 to 160 for A-B

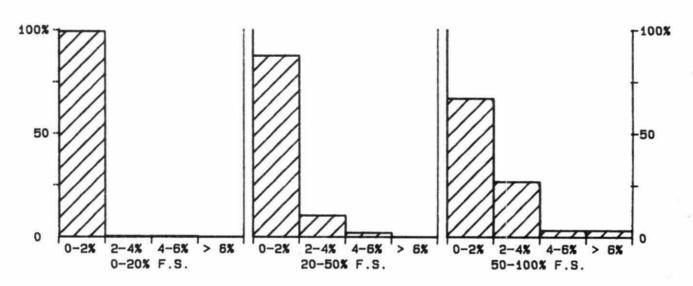
DUPL ICATES:	Number of	Sample			Mean(2) s.d.	Coefficient
	Data Pairs	Concn Span		of var.(%)		
	208	Ø	-	25	1.1	11.1
	46	25	-	50	2.0	5.4
	47	50	-	125	2.6	3.4
	23	125	-	250	4.9	2.8
	324	04	era	11	2.0	N/A

DETECTION CRITERION: 3

## QUALITY CONTROL GRAPHS SULPHATE (UG/FILTER AS SO4)

FROM: 04/01/85 TO: 27/12/85





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 250 UG/FILTER AS SO4

### *** SULPHATE ***

### IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80

LIS Test Name Code: SSO4UR Units : ug/Filter as SO4

Work Station Code : PRLOV Unit Code : 361941

Method Code : 004AIC Supervisor : M. Rawlings

Sample Type/Matrix: W40 filters from LoVol filter packs.

### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO4.

Full scale conductivity : 30 uS/cm.

N.B. Nitrate and chloride are determined simultaneously.

### INSTRUMENTATION:

-Ultrasonic bath; polyethylene tubes

-Automated modular continuous flow ion chromatographic system.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 2.50

Detection Criterion (T): 9

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

### MODIFICATIONS:

01/08/81 - Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

10/05/85 - Microcomputer used for data reduction.

10/05/85 - Three additional calibration standards were set up.

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

No data summary available for period not covered in performance report.

### SULPHATE QUALITY CONTROL DATA FROM 05/02/85 TO 10/12/85

Lab: Precipitation

Analytical Range: 9 to 500 ug/Filter as SO4

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	26	400	401	1	3.6
b	1	26	100	99	-1	1.7
a+b	:	26	500	500	Ø	3.4
a-b		26	300	302	2	4.4

s.d.(AB): Sw(within run): 3.1 S(between runs):

2.8 S/Sw: 0.90

On any given day the calibration is accepted if the values obtained lie within the ranges:

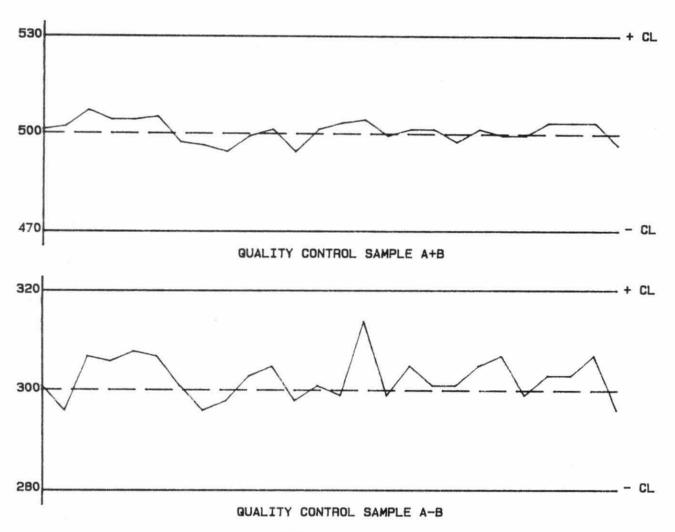
470 to 530 for A+B to 320 for A-B 280

DUPL I CATES :	Number of	Sample		Mean(2)	Coefficient	
	Data Pairs	Conc	n 8	pan	s.d.	of var.(%)
	20	Ø	-	50	3.1	14.4
	10	50	-	100	3.8	4.6
	6	100	-	250	2.8	1.9
	7	250	-	500	2.9	0.9
	43	04	era	11	3.2	N/A

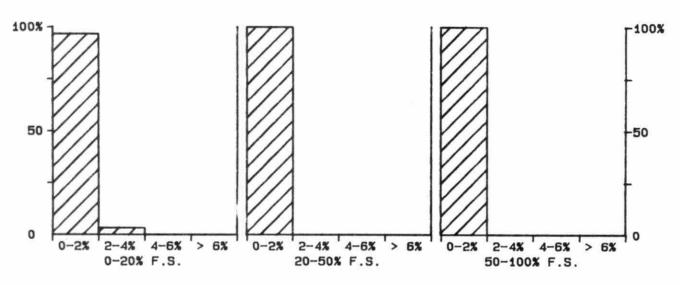
DETECTION CRITERION: 9

# QUALITY CONTROL GRAPHS SULPHATE (UG/FILTER AS SO4)

FROM: 05/02/85 TO: 10/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 500 UG/FILTER AS SO4

### *** SULPHATE ***

### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/82
LIS Test Name Code: SSO4UR Units : mg/L as SO4
Work Station Code : RMDSO4 Unit Code : 064941
Method Code : 003AI0 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as 804 is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 100 uS/cm.

### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module(in-house design) for automated sample introduction and timing.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.05

Detection Criterion (T): 0.76

### CALIBRATION:

BL plus 16 standards

### CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : 2 standards

### MODIFICATIONS:

01/04/82- The sulphate procedure that was introduced differed slightly from Method B in HAMES: (1) full scale values for the analytical ranges were 20.0 and 50.0 mg/L, and (2) samples were not spiked with concentrated eluent. The latter was not necessary because only sulphate was measured and spiking is required for chloride analysis.

01/01/84- Packed suppressor column was replaced by a fibre suppressor(walls of fibre are ion-exchange media). Full scale for high analytical was increased from 50.0 to 100 mg/L as SO4; QC standards were adjusted accordingly. Analytical rate was doubled.

17/10/85- Increase number of standards to 16 to ensure proper calibration at low end of analytical range.

# SULPHATE QUALITY CONTROL DATA FROM 02/01/85 TO 23/12/85

Lab: Rivers and Lakes

Analytical Range: 0.76 to 100.0 mg/L as SO4

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	110	60.0	60.0	-0.0	0.78
b		110	15.0	15.2	0.2	0.39
a+	b :	110	75.0	75.2	0.2	0.90
a -	b :	110	45.0	44.8	-0.2	0.83
c	:	111	15.00	15.04	0.04	0.203
d	:	111	5.00	5.05	0.05	0.108
c +	d :	111	20.00	20.09	0.09	0.225
c -	d :	111	10.00	10.00	-0.00	0.236

s.d.(AB): Sw(within run): 0.59 S(between runs): 0.62 S/Sw: 1.05 s.d.(CD): Sw(within run): 0.167 S(between runs): 0.163 S/Sw: 0.97

In any given day the calibration is accepted if the values obtained lie within the ranges:

70.5 to 79.5 for A+B 42.0 to 48.0 for A-B 19.10 to 20.30 for C+D 9.40 to 10.60 for C-D

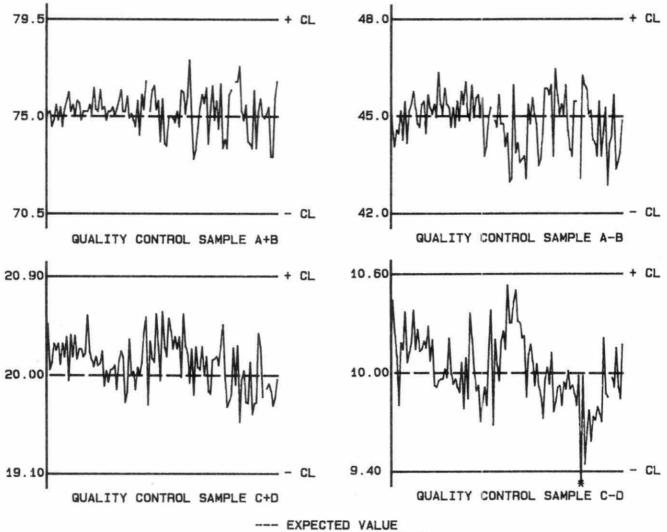
DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	15	0.00 - 2.00	0.253	29.8
	38	2.00 - 5.00	0.178	4.8
	189	5.00 - 10.00	0.235	3.2
	72	10.0 - 20.0	0.52	3.9
	49	20.0 - 100.0	1.07	2.7
	363	Overal1	0.49	N/A

### DETECTION CRITERION: 0.76

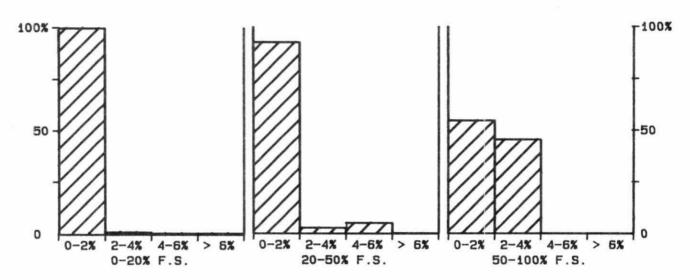
THER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	1	112	0.00	0.020

## QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 02/01/85 TO: 23/12/85



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS SO4

#### SULPHUR DIOXIDE *** * * *

### IDENTIFICATION:

: Precipitation Method Introduced: 01/07/80

LIS Test Name Code: SSO2FR Units : ug/Filter as SO2

Work Station Code : PRSEQ, PRLOV Unit Code : 361943

Method Code : 004AI0 Supervisor : M. Rawlings

Sample Type/Matrix: Impregnated W41 filters from sequential and LoVol filter packs.

### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

Other : Filter is impregnated with potassium carbonate/glycerol

solution.

### SAMPLE PREPARATION:

Filters are extracted with 50 ml. of 0.05% H202 in polystyrene tubes with 1 hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period. SO2 is converted to SO4 in the process.

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO2.

Full scale conductivity : 10 uS/cm.

### INSTRUMENTATION:

-Mechanical shaker; ultrasonic bath; polyethylene tubes

-Automated modular continuous flow ion chromatographic system.

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 1.65

Detection Criterion (T): 3.9

### CALIBRATION:

BL plus 3 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

### MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified for analysis of W41 filter extracts by developing the extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

15/03/84 - Streamlined procedure for extraction of W41 filters in one 50 mL polyethylene tube was adopted, eliminating two container transfers, and changing the extraction volume to 50.0 mL from 100.0 mL. Full scale reduced from 700 to 350ug/filter as SO2.

10/05/85 - Microcomputer used for data reduction.

- Three additional calibration samples were set up.

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

### SULPHUR DIOXIDE QUALITY CONTROL DATA FROM 04/01/85 TO 27/12/85

Lab: Precipitation

Analytical Range: 3.9 to 350 ug/Filter as SO2

CAL	TODAT	PTIBLE	COL	TROL:
LITE	IBKH	NULL	LUN	IIKUL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	1	146	268	265	-3	3.5
b		146	66	67	1	2.1
a+b	1	146	334	333	-1	4.3
a-b		146	202	198	-4	3.9

s.d.(AB): Sw(within run): 2.8 S(between runs): 2.9 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

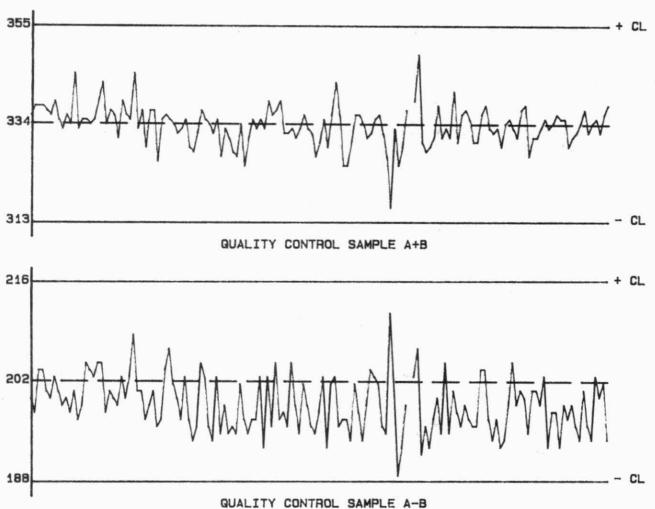
> 313 to 355 for A+B 188 to 216 for A-B

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	179	0.0 - 35.0	1.28	15.0
	40	35.0 - 70.0	3.18	5.8
	45	70.0 - 175.0	5.01	4.1
	16	175 - 350	10.6	4.4
	280	Overall	3.6	N/A

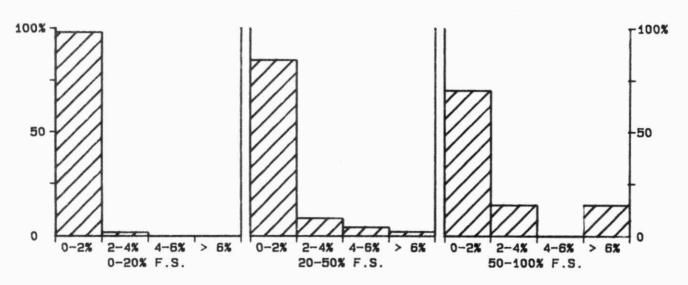
DETECTION CRITERION: 3.9

# QUALITY CONTROL GRAPHS SULPHUR DIOXIDE (UG/FILTER AS SO2)

FROM: 04/01/85 TO: 27/12/85



-- EXPECTED VALUE - CONTROL LIMIT (CL) DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 350 UG/FILTER AS SO2

### *** TURBIDITY ***

IDENTIFICATION:

Laboratory : Domestic Water Method : Before '74

LIS Test Name Code: TURB Units : FTU
Work Station Code: WTURB Unit Code : 343000

Method Code : 002AII Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Water, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

The instrument is standarized periodically with freshly prepared formazin standards. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

Hach Ratio 18900 Turbidimeter

REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.09

### CALIBRATION:

BL plus formazin standards (at least once annually)

### CONTROLS:

Calibration: Bl plus two standards, eg, QCA *

### MODIFICATIONS:

01/03/84-Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter. In the past samples were not stirred during turbidity measurements in the Domestic Water laboratory even though the former instrument (Hach 2100A) possessed this capability. Thus the effect of changing the instrumentation was minimal.

01/09/85 Controls QCA,QCB introduced: These controls are aqueous suspensions of beads composed of a styrene-divinylbenzene polymer and are formulated to "match" the performance of formazin standards on the HACH 18900 turbidimeter.
*Insufficient data collected for inclusion in performance report.

### TURBIDITY QUALITY CONTROL DATA FROM 03/01/85 TO 30/12/85

Lab: Domestic Water Analytical Range: 0.09 to 200 FTU DUPL I CATES! Number of Sample Mean(2) Coefficient Concn Span Data Pairs s.d. of var.(%) ----------0.00 - 2.00 181 0.031 4.8 88 2.0 - 20.0 0.39 5.8 20 - 100 100 - 200 17

Overall

0.8

0.8

0.3

2.0

0.5

N/A

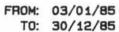
DETECTION CRITERION: 0.09

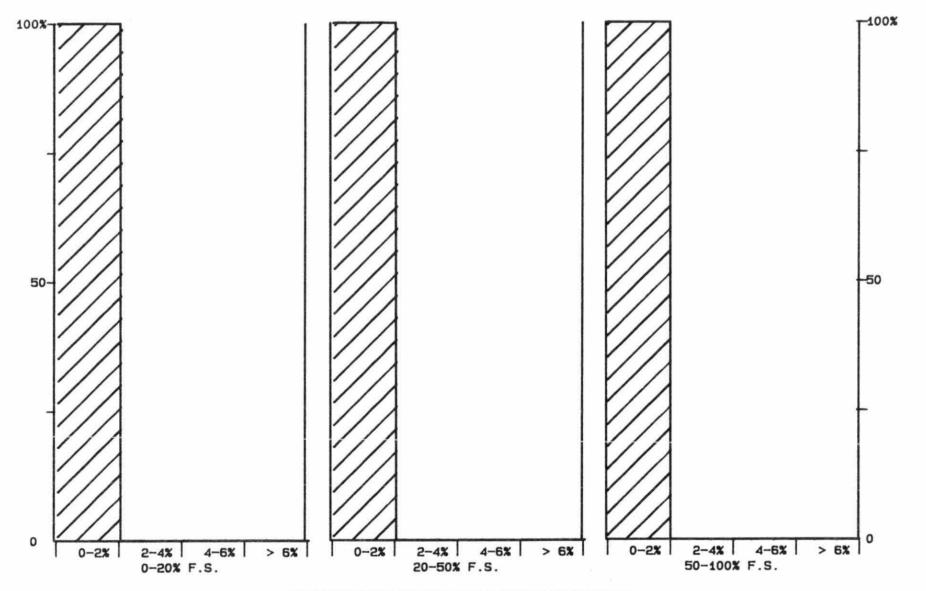
6

292

# 27

# QUALITY CONTROL GRAPH TURBIDITY (FTU)





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 200 FTU

### *** TURBIDITY ***

### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74

LIS Test Name Code: TURB Units : FTU
Work Station Code: RMTURB Unit Code : 343000

Method Code : 002AII Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

The instrument is standarized with a sealed standard which is prepared commercially from latex polymers of Known size and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

### INSTRUMENTATION:

- Hach Ratio 18900 Turbidimeter

### REPORTING:

Maximum Significant Figures: 3 Minimum Increment (W): 0.01

Detection Criterion (T): 0.21

### CALIBRATION:

BL plus formazin standards (at least once annually)

### CONTROLS:

Calibration: Bl plus two standards, eg, QCA*

### MODIFICATIONS:

01/04/82-Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter. As of this date samples are no longer stirred during turbidity measuremeants, and thus the effect of heavy particulates is minimized as they settle out before the reading is accepted.

01/09/85-Controls QCA, QCB introduced: these controls are aqueous suspensions of beads composed of a styrene-divinylbenzene polymer and are formulated to "match" the performance of formazin standards on the Hach 18900 turbidimeter.

*Insufficient data collected for inclusion in performance report.

# TURBIDITY QUALITY CONTROL DATA FROM 03/01/85 TO 02/10/85

Lab: Rivers and Lakes

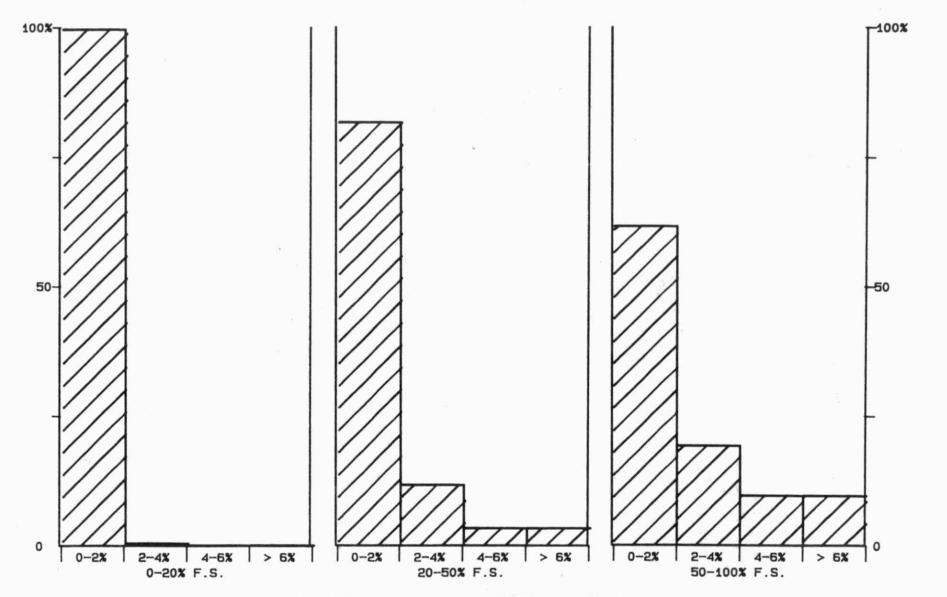
Analytical Range: 0.21 to 50.0 FTU

DUPL I CATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	78	0.00 - 2.00	0.069	5.0
	110	2.00 - 5.00	0.166	4.9
	74	5.00 - 10.00	0.219	3.0
	90	10.0 - 50.0	1.17	5.5
	352	Overall	0.61	N/A

DETECTION CRITERION: 0.21

# QUALITY CONTROL GRAPH TURBIDITY (FTU)

FROM: 03/01/85 TO: 02/10/85



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 50 FTU

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